10/731,733

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 NEWS  8  DEC 15  MEDLINE update schedule for December 2004
 NEWS  9  DEC 17  ELCOM reloaded; updating to resume; current-awareness
                  alerts (SDIs) affected
 NEWS  10 DEC 17  COMPUAB reloaded; updating to resume; current-awareness
                  alerts (SDIs) affected
 NEWS  11 DEC 17  SOLIDSTATE reloaded; updating to resume; current-awareness
                  alerts (SDIs) affected
 NEWS  12 DEC 17  CERAB reloaded; updating to resume; current-awareness
                  alerts (SDIs) affected
 NEWS  13 DEC 17  THREE NEW FIELDS ADDED TO IFIPAT/IFIUDB/IFICDB
 NEWS  14 DEC 30  EPFULL: New patent full text database to be available on STN
 NEWS  15 DEC 30  CAPLUS - PATENT COVERAGE EXPANDED
 NEWS  16 JAN 03  No connect-hour charges in EPFULL during January and
                  February 2005
 NEWS  17 JAN 11  CA/CAPLUS - Expanded patent coverage to include Russia
                  (Federal Institute of Industrial Property)

 NEWS EXPRESS  JANUARY 10 CURRENT WINDOWS VERSION IS V7.01a, CURRENT
               MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),
               AND CURRENT DISCOVER FILE IS DATED 10 JANUARY 2005

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=> fil casreact
COST IN U.S. DOLLARS

	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	0.21	0.21

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This file contains CAS Registry Numbers for easy and accurate substance identification.

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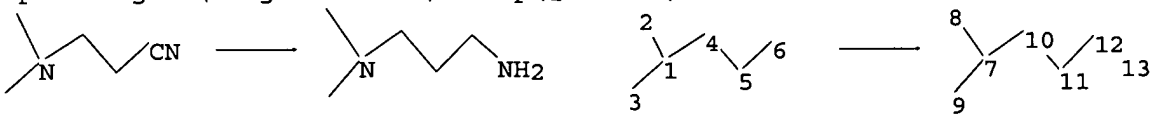
Uploading C:\Program Files\Stnexp\Queries\10731733.str



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1 2 3 4 5 6 7 8 9 10 11 12 13
chain bonds :
1-2 1-3 1-4 4-5 5-6 7-8 7-9 7-10 10-11 11-12 12-13
exact/norm bonds :
1-2 1-3 1-4 7-8 7-9 7-10 12-13
exact bonds :
4-5 5-6 10-11 11-12

Match level :
1:CLASS 2:CLASS 3:CLASS 4:CLASS 5:CLASS 6:CLASS 7:CLASS 8:CLASS 9:CLASS 10:CLASS 11:CLASS 12:CLASS 13:CLASS
fragments assigned product role:
containing 7
fragments assigned reactant/reagent role:
containing 1

L1 STRUCTURE UPLOADED

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=> d query
L1                STR
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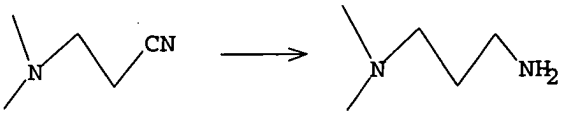


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Structure attributes must be viewed using STN Express query preparation.


=> s l1
SAMPLE SEARCH INITIATED 17:35:04 FILE 'CASREACT'
SCREENING COMPLETE -     140 REACTIONS TO VERIFY FROM      40 DOCUMENTS

100.0% DONE     140 VERIFIED        9 HIT RXNS                        6 DOCS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS:  ONLINE  **COMPLETE**
                        BATCH   **COMPLETE**
PROJECTED VERIFICATIONS:       2091 TO     3509
PROJECTED ANSWERS:                6 TO      266

L2             6 SEA SSS SAM L1 (      9 REACTIONS)

=> s l1 full
FULL SEARCH INITIATED 17:35:09 FILE 'CASREACT'
SCREENING COMPLETE -    4617 REACTIONS TO VERIFY FROM     901 DOCUMENTS

100.0% DONE    4617 VERIFIED      139 HIT RXNS                       70 DOCS
SEARCH TIME: 00.00.01

L3            70 SEA SSS FUL L1 (    139 REACTIONS)

=> fil caplus
COST IN U.S. DOLLARS                                SINCE FILE      TOTAL
                                                       ENTRY    SESSION
FULL ESTIMATED COST                                   106.53     106.74

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FILE COVERS 1907 - 21 Jan 2005  VOL 142 ISS 5
FILE LAST UPDATED: 20 Jan 2005  (20050120/ED)

  This file contains CAS Registry Numbers for easy and accurate
  substance identification.
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=> s l3
L4           70 L3

=> s ni or nickel
         579947 NI
           3697 NIS
         582181 NI
                  (NI OR NIS)
         567392 NICKEL
            193 NICKELS
         567420 NICKEL
                  (NICKEL OR NICKELS)
L5       787048 NI OR NICKEL

=> s co or cobalt
         781010 CO
          26015 COS
         803596 CO
                  (CO OR COS)
         345184 COBALT
             95 COBALTS
         345187 COBALT
                  (COBALT OR COBALTS)
L6       938306 CO OR COBALT

=> s l4 and l5
L7           16 L4 AND L5

=> s l4 and l6
L8            9 L4 AND L6

=> s l7 or l8
L9           20 L7 OR L8

=> d l9 1-20 abs ibib
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L9 ANSWER 1 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN

AB A low-pressure hydrogenation process for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile in the presence of a catalyst system comprising sponge **nickel** and aqueous alkali is described.

ACCESSION NUMBER: 2004:609968 CAPLUS
DOCUMENT NUMBER: **141:140075**
TITLE: Low-pressure hydrogenation process for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile in the presence of a catalyst system comprising sponge **nickel** and aqueous alkali
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): USA
SOURCE: U.S. Pat. Appl. Publ., 8 pp., Cont.-in-part of U.S. Ser. No. 327,765.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004147784	A1	20040729	US 2003-731733	20031209
US 6660887	B1	20031209	US 2002-327765	20021223
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-327765 A2 20021223
US 2003-731733 A 20031209

OTHER SOURCE(S): CASREACT 141:140075

L9 ANSWER 2 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN

AB A low-pressure hydrogenation process for the production of 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile (I) comprises: feeding hydrogen and 3-(dimethylamino)propionitrile into a low-pressure reactor containing a sponge **nickel** catalyst, at least one Group IA alkali metal hydroxide (e.g., potassium hydroxide), and water to form a reaction medium; heating the reaction medium to 70-100°; pressurizing the reactor to 45-500 psig; and hydrogenating the nitrile to form I.

ACCESSION NUMBER: 2004:589527 CAPLUS
DOCUMENT NUMBER: **141:123405**
TITLE: Low-pressure catalytic hydrogenation process for the manufacture of 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): Solutia Inc., USA
SOURCE: PCT Int. Appl., 20 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
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US 6660887	B1	20031209	US 2002-327765	20021223
US 2004147784	A1	20040729	US 2003-731733	20031209

PRIORITY APPLN. INFO.: US 2002-327765 A 20021223
US 2003-731733 A 20031209

OTHER SOURCE(S): CASREACT 141:123405

L9 ANSWER 3 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN

AB A process for the production of 3-(dimethylamino)propylamine (I) in high (>99%) purity from 3-(dimethylamino)propionitrile utilizing a low-pressure hydrogenation process is described which comprises contacting the nitrile with hydrogen at low pressure in the presence of a sponge **nickel** catalyst and ≥1 Goup IA metal hydroxide at 70-100°/45-150 psig. The improvement in the process resides in a combination of carrying out the hydrogenation process at low pressures and temps. in the presence of a catalytic amount of caustic base in order to give a I selectivity of >99.60%.

ACCESSION NUMBER: 2003:961180 CAPLUS
DOCUMENT NUMBER: **140:17730**
TITLE: Low-pressure hydrogenation process and catalyst system for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): Solutia Inc., USA
SOURCE: U.S., 7 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6660887	B1	20031209	US 2002-327765	20021223
WO 2004060039	A2	20040722	WO 2003-US29721	20030919
WO 2004060039	A3	20040826		

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 2004147784	A1	20040729	US 2003-731733	20031209
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-327765 A 20021223
US 2003-731733 A 20031209

L9 ANSWER 3 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

OTHER SOURCE(S): CASREACT 140:17730
REFERENCE COUNT: 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 4 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN

AB Primary amines were prepared by hydrogenation of nitriles in the presence of catalysts containing Co and optionally Ni as well as ≥1 doping metal on a particulate substrate, whereby the Co and optional Ni have an avg. particle size of 3-30 nM. Thus, dimethylaminopropionitrile was hydrogenated in the presence of a suspension catalyst [prepared from Co(NO3)2, Ni(NO3)2, and Y(NO3)3 and aluminosilicate powder] at 80° in the presence of NH3 and 80 bar H2 to give dimethylaminopropylamine in 98.4% selectivity.

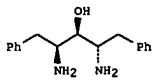
ACCESSION NUMBER: 2003:332011 CAPLUS
DOCUMENT NUMBER: 138:337704
TITLE: Preparation of primary amines via reduction of nitriles in the presence of supported cobalt catalysts containing dopants and optionally nickel.
INVENTOR(S): Ansmann, Andreas; Benisch, Christoph
PATENT ASSIGNEE(S): BASF AG, Germany
SOURCE: Ger. Offen., 14 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10152135	A1	20030430	DE 2001-10152135	20011023
US 2003120115	A1	20030626	US 2002-271977	20021017
US 6790996	B2	20040914		
EP 1306365	A2	20030502	EP 2002-23640	20021021
EP 1306365	A3	20031015		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK				
JP 2003192647	A2	20030709	JP 2002-307884	20021023
PRIORITY APPLN. INFO.:			DE 2001-10152135 A	20011023

OTHER SOURCE(S): CASREACT 138:337704; MARPAT 138:337704

L9 ANSWER 5 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN

GI



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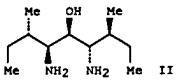

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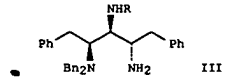

III

AB Nonracemic pseudo-C2-sym. diamino alc. and triamines are prepared stereoselectively from amino acid-derived aldehydes as potential fragments for HIV protease inhibitors. Addition of organolithium reagents to siloxy nitriles derived from L-phenylalaninal and L-isoleucinal, followed by in situ reduction of the intermediate imines and transfer hydrogenation and deprotection of N-benzyl protecting groups under microwave irradiation, led to 1,3-diamino alcs. such as I and II in moderate to good yields. Nonracemic pseudo-C2-sym. triamine III (R = 4-MeOC6H4) is prepared using a scandium triflate-catalyzed nitro-Mannich addition of the silylnitronate PhCH2CH:N+(O-)OSiMe3 derived from 2-phenyl-1-nitroethane to (S)-PhCH2CH(NBn2)CH:N-4-C6H4OMe, derived from L-phenylalaninal; reduction of the nitro group with sodium borohydride and nickel (II) chloride, and separation of diastereomers yields III (R = 4-MeOC6H4).

ACCESSION NUMBER: 2003:50024 CAPLUS
DOCUMENT NUMBER: 138:237779
TITLE: Concise and Stereocontrolled Synthesis of Pseudo-C2-symmetric Diamino Alcohols and Triamines for Use in HIV Protease Inhibitors
AUTHOR(S): Bernardi, Luca; Bonini, Bianca F.; Dessole, Gabriella; Fochi, Mariafrancesca; Comes-Franchini, Mauro; Gavioli, Silvia; Ricci, Alfredo; Varchi, Greta
CORPORATE SOURCE: Dipartimento di Chimica Organica "A. Mangini", Facolta di Chimica Industriale, Universita di Bologna, Bologna, 40136, Italy
SOURCE: Journal of Organic Chemistry (2003), 68(4), 1418-1425
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal

L9 ANSWER 5 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:237779
REFERENCE COUNT: 43 THERE ARE 43 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 6 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN

AB Primary amines are prepared by catalytic hydrogenation of nitriles in the presence of W-containing Raney Ni catalysts. Ni-Al-W alloy (43:56:1) was heated in aqueous NaOH at 95-100° for 2 h to give a catalyst, which was used in hydrogenation of 3-[N-(2-hydroxyethyl)-N-methylamino]propionitrile in the presence of aqueous NH3 at 65° under 2.0 MPa for 2.0 h to give 97% N-(2-hydroxyethyl)-N-methyl-1,3-propanediamine.

ACCESSION NUMBER: 2001:496310 CAPLUS
DOCUMENT NUMBER: 135:92364
TITLE: Preparation of primary amines and catalysts for reduction of nitriles
INVENTOR(S): Kikuchi, Ryuji; Nagai, Naofumi; Arakawa, Tatsuya
PATENT ASSIGNEE(S): Kawaken Fine Chemicals Co., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2001187766	A2	20010710	JP 2000-123106	20000424
PRIORITY APPLN. INFO.:			JP 1999-301544 A	19991022

OTHER SOURCE(S): CASREACT 135:92364

L9 ANSWER 7 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN
AB Ni(II) complexes with the aliphatic tripodal tetraamine ligands N(CH2CH2NH2)3 (tren, 1), N[(CH2CH2CH2NH2)(CH2CH2NH2)2] (baep, 2), N[(CH2CH2CH2NH2)2(CH2CH2NH2)] (abap, 3), and N(CH2CH2CH2NH2)3 (trpn, 4) are reported. The tripodal tetradentate N4 ligands 1-4 react with Ni(NO3)2·6H2O in MeCN or MeOH to give the blue Ni (II) complexes [Ni(1)(η1-NO3)2] (5a), [Ni (2)(η1-NO3)2] (5b), [Ni(3)(η2-NO3)]NO3·MeCN (5c·MeCN), and [Ni(4)(η2-NO3)]NO3·MeOH (5d·MeOH). With NiCl2·6H2O, [Ni (1)Cl(H2O)]Cl·H2O (6a), {Ni(2)(μ-Cl)2Ni(2)}Cl2·2MeOH (6b·2MeOH), [Ni(3)Cl(H2O)]Cl (6c), and {Ni(4)(H2O)2}Cl2·H2O (6d·H2O) were obtained. The mol. structures of 5a-d and 6b-d were determined by x-ray diffraction anal. and they are compared with the mol. structure of the previously characterized complex 6a. Complexes 5a-d and 6b-d exhibit octahedrally coordinated Ni atoms. The tripodal ligands occupy four of the six coordination sites in a pseudo-facial manner. Complexes of the unsym. 2 and 3 possess both five- and six-membered chelate rings. The extension of the ligand arms in 1-4 leads to a systematic variation in the geometric and UV/visible spectroscopic properties of the complexes depending on the size of the chelate rings formed by the ligands.
ACCESSION NUMBER: 2001:335178 CAPLUS
DOCUMENT NUMBER: **135:115961**
TITLE: Cis-octahedral **nickel**(II) complexes with symmetric and unsymmetric tripodal tetraamine ligands
AUTHOR(S): Ochs, Christian; Hahn, F. Ekkehardt; Lugger, Thomas
CORPORATE SOURCE: Institut fur Anorganische und Analytische Chemie der Freien Universitat Berlin, Berlin, 14195, Germany
SOURCE: European Journal of Inorganic Chemistry (2001), (5), 1279-1285
CODEN: EJICFO; ISSN: 1434-1948
PUBLISHER: Wiley-VCH Verlag GmbH
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 135:115961
REFERENCE COUNT: 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE
FORMAT

L9 ANSWER 8 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN
AB Nitriles NCCH2CH2NX-R-NXCH2CH2CN [R = (CH2)n, 1,2-cyclohexyl, 1,4-cyclohexyl, CHMeCH2, X = Boc, n = 4, 7, 9, 12; R = 1,2-Ph, 1,3-Ph, 1,4-Ph, X = H] are reduced to primary amines in the presence of N-tert-butoxycarbonyl (Boc) groups. E.g., NCCH2CH2N(Boc)(CH2)4N(Boc)CH2CH2CN in absolute ethanol and THF was stirred with a mixture of Pd/C and Raney **nickel** and sodium hydroxide; the mixture was shaken for 8 h under 45 psi of hydrogen and worked up to give H2N(CH2)3N(Boc)(CH2)4N(Boc)(CH2)3NH2 in 84% yield. The reduction can be carried out under atmospheric H2 pressure using com. avail. catalysts. Both Boc groups and aromatic moieties present in the starting material are well tolerated under the mild optimized conditions.
ACCESSION NUMBER: 2001:156286 CAPLUS
DOCUMENT NUMBER: **134:326005**
TITLE: Nitrile reduction in the presence of Boc-protected amino groups by catalytic hydrogenation over palladium-activated Raney-**nickel**
AUTHOR(S): Klenke, Burkhard; Gilbert, Ian H.
CORPORATE SOURCE: Welsh School of Pharmacy, Cardiff University, Cardiff, CF10 3XF, UK
SOURCE: Journal of Organic Chemistry (2001), 66(7), 2480-2483
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:326005
REFERENCE COUNT: 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE
FORMAT

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GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Starting from aspartic acid, the authors synthesized lactam-bridged β- and γ-amino acid equivalent Using the 1,4-bis electrophile (PhCH2)2NCH(CH2OSO2Me)CH2CH2OSO2Me as a central intermediate, the 4- and 5-aminopiperidin-2-ones I and II (R = H, Me3COCO, PhCH2) were prepared by regioselective functionalization and subsequent lactamization. Diastereoselective C-alkylation was performed after N-protection of the lactam functionality when exclusive trans configuration resulting in the formation of III (R = Me3COCO, CH2Ph; R1 = Me, PhCH2, F) was observed in the 4-amino series. On the other hand, cis selectivity was typical for the alkylations of the 5-amino lactams III (R = Boc; R1 = Me, PhCH2). To investigate the ability of the lactam building blocks to induce reverse-turn structures by intramol. hydrogen bonding, the model peptidomimetics IV and V representing Homo-Freidinger lactams were prepared from I and II (R = H), resp. Conformational analyses in dilute solution (1 mM) by IR and NMR spectroscopy at room temperature clearly indicated that the 4-aminopiperidin-2-one derivative IV predominantly adopts a reverse-turn structure stabilized by a CO-HN hydrogen bond in an 11-membered ring. VT NMR expts. showed a substantial temperature dependency of the terminal NH when Δδ NH/ΔT = -6.5 indicated that the amount of intramol. hydrogen bonding is higher at low temperature An application in the field of medicinal chemical was demonstrated. Thus, starting from a Homo-Freidinger lactam and its enantiomer, the authors synthesized the peptidomimetics (2S,4S)-VI and (2S,4R)-VI and investigated them as lactam-bridged analogs of the dopamine receptor modulating peptide Pro-Leu-Gly-NH2 (PLG). Both test compds. turned out to enhance significantly the agonist binding of dopamine D2 receptors, when the isomer 15c revealed a potency comparable to the genuine ligand PLG.
ACCESSION NUMBER: 2000:692927 CAPLUS
DOCUMENT NUMBER: **134:17379**
TITLE: Enantiopure 4- and 5-aminopiperidin-2-ones: regiocontrolled synthesis and conformational characterization as bioactive β-turn mimetics
AUTHOR(S): Weber, Klaus; Ohnmacht, Ursula; Gmeiner, Peter
CORPORATE SOURCE: Department of Medicinal Chemistry Emil Fischer Center, Friedrich-Alexander University, Erlangen, D-91052, Germany
SOURCE: Journal of Organic Chemistry (2000), 65(22), 7406-7416
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:17379
REFERENCE COUNT: 46 THERE ARE 46 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE

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AB Complexes between **cobalt**(III) and eight different 1,4,7,10-tetraazacyclododecane (cyclen) as well as two tris(3-aminopropyl)amine (trpn) derivs. are reported with varying nos. and structures of peralkylammonium groups in side chains of the ligands. The presence of addnl. pos. charges has small effects on hydrolysis rates of nitrophenyl- and bis(nitrophenyl)phosphate esters but leads to substantially enhanced cleavage of plasmid DNA. Increasing the number of the charged side groups and/or their distance to the metal ion center provides for better binding to the DNA groove, as shown also by affinity measurements with calf-thymus DNA. In line with this, saturation kinetics of plasmid DNA cleavage yield a corresponding increase of efficiency in Michaelis-Menten-type KM values, with rather constant kcat parameters. A binuclear **cobalt** complex with two cyclen centers separated by a -(CH2)6-N+(CH3)2-(CH2)6-N+(CH3)2-(CH2)6- spacer shows, with only 5+10-5 M catalyst concentration, the largest known rate enhancement factor of >107 (corresponding to >1011 at 1 M) against DNA; incubation with 0.05 mM at 37° for only 2 h leads to almost complete cleavage without appearance of products typical for redox cleavage. These results are in contrast to expts. with corresponding copper(II) complexes with added hydrogen peroxide, which has no effect with corresponding Co, Zn, Cd, or Ni complexes.

ACCESSION NUMBER: 1997:347191 CAPLUS
DOCUMENT NUMBER: **127:46819**
TITLE: **Cobalt**(III) Polyamine Complexes as Catalysts for the Hydrolysis of Phosphate Esters and of DNA. A Measurable 10 Million-Fold Rate Increase
AUTHOR(S): Hettich, Ronald; Schneider, Hans-Joerg
CORPORATE SOURCE: FR Organische Chemie der Universitaet des Saarlandes, Saarbruecken, D 66041, Germany
SOURCE: Journal of the American Chemical Society (1997), 119(24), 5638-5647
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 127:46819
REFERENCE COUNT: 64 THERE ARE 64 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

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AB RNR1(CH2)3NH2 [R, R1 = H, C1-22 (hydroxy)alkyl] are prepared by catalytic hydrogenation of RNR1(CH2)2CN in the presence of RNHR1. Autoclaving a mixture of 3-[N-(2-hydroxyethyl)-N-methylamino]propionitrile, MeNHCH2CH2OH, and Raney **Ni** at 60° and 30 kg/cm2-gage for 3 h to give 89% N-(2-hydroxyethyl)-N-methyl-1,3-propanediamine.

ACCESSION NUMBER: 1995:780699 CAPLUS
DOCUMENT NUMBER: **123:339135**
TITLE: Preparation of N-(3-aminopropyl)amines
INVENTOR(S): Kaita, Jun; Ootawa, Yasunori; Kato, Tooru; Sotodani, Koshiro
PATENT ASSIGNEE(S): Kao Corp, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 07157453	A2	19950620	JP 1993-302605	19931202
JP 3224922	B2	20011105		
PRIORITY APPLN. INFO.:			JP 1993-302605	19931202

OTHER SOURCE(S): CASREACT 123:339135; MARPAT 123:339135

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AB HOCnH2nNR(CH2)3NH2 [I; R = H, C1-6 (hydroxy)alkyl; n = 2-9], useful as materials for surfactants, softeners, dyes, acidic gas removers, polymers, etc. (no data), are prepared by reduction of HOCnH2nNR(CH2)2CN (II; R, n = same as I) by H in presence of Raney **Ni**, NH3, and 0-50 weight% (based on II) C1-5 alcs. II (R = Me, n = 2) was treated with Raney **Ni** and NH3 under 20 kg/cm2-G H at 55-65° for 5 h to give 92% I (R = Me, n = 2).

ACCESSION NUMBER: 1994:163451 CAPLUS
DOCUMENT NUMBER: **120:163451**
TITLE: Preparation of N-(3-aminopropyl)amines from amino nitriles
INVENTOR(S): Tatezawa, Osamu; Kitayama, Hiroaki; Kaita, Jun; Kato, Tooru; Sotodani, Koshiro
PATENT ASSIGNEE(S): Kao Corp, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 3 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05246959	A2	19930924	JP 1992-46779	19920304
JP 2951790	B2	19990920		
PRIORITY APPLN. INFO.:			JP 1992-46779	19920304

OTHER SOURCE(S): CASREACT 120:163451; MARPAT 120:163451

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AB Title compds. were prepared by Michael addition of polyamines with acrylonitrile followed by reduction The reduction of polynitriles is achieved by the use of Dibal in high yield. Thus, N(CH2CH2NH2)3 was refluxed 24h in CH2:CHCN containing HOAC to give 81% N[CH2CH2N(CH2CH2CN)2]3 which was refluxed 24h with Dibal in THF/hexane to give 90% N[CH2CH2N(CH2CH2NH2)2]3 (I). I was further condensed with acrylonitrile. Condensation of I with 2-(HO)C6H4CHO gave a dendrimeric hexaimine which was used to form a tricobalt complex.

ACCESSION NUMBER: 1994:163403 CAPLUS
DOCUMENT NUMBER: **120:163403**
TITLE: Preparation of dendrimeric polyamines
AUTHOR(S): Moors, Rolf; Voegtle, Fritz
CORPORATE SOURCE: Inst. Org. Chem. Biochem., Univ. Bonn, Bonn, D-53121, Germany
SOURCE: Chemische Berichte (1993), 126(9), 2133-5
CODEN: CHBEAM; ISSN: 0009-2940
DOCUMENT TYPE: Journal
LANGUAGE: German
OTHER SOURCE(S): CASREACT 120:163403

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AB The title compound (I), useful as a chain extender for polyurethanes, is prepared with high yield, selectivity, and purity by adding acrylonitrile (II) to HCONH2 in the presence of 4-aminopyridine derivs. and hydrogenating the resulting dinitrile. Refluxing II 212, HCONH2 180, 4-(dimethylamino)pyridine 14.7, and MeCN 212 g for 21 h gave 170 g HCON(CH2CH2CN)2 (III) with purity 99% and selectivity 95%. Hydrogenating 255 g III in MeOH-NH3 over 40 g Raney Fe-Ni (15:85) at 30°/30-50 bar gave 262 g I (.apprx.50:50 mixture of N1 and N2 isomers).

ACCESSION NUMBER: 1990:119575 CAPLUS
DOCUMENT NUMBER: **112:119575**
TITLE: Bis(trimethylene)triamine monoformamide manufacture
INVENTOR(S): Scholl, Hans Joachim; Reiff, Helmut
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.
SOURCE: Ger. Offen., 6 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3811342	A1	19891012	DE 1988-3811342	19880402
CA 1309728	A1	19921103	CA 1989-593838	19890315
EP 336184	A1	19891011	EP 1989-104900	19890318
EP 336184	B1	19930113		
R: CH, DE, FR, GB, IT, LI, NL				
US 4923952	A	19900508	US 1989-329472	19890328
JP 02006444	A2	19900110	JP 1989-78757	19890331
JP 2583450	B2	19970219		

PRIORITY APPLN. INFO.: DE 1988-3811342 A 19880402

OTHER SOURCE(S): CASREACT 112:119575; MARPAT 112:119575

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AB Me2N(CH2)3NH2 (I) is prepared by hydrogenation of Me2NCH2CH2CN (II) in the presence of an alkaline earth oxide which suppresses formation of H2N(CH2)3NH2 (III). Thus, II, Raney Co, and NH3 (liquid) were charged to an autoclave and the whole maintained at 160° and 150 bar H to give I containing <50 ppm III.

ACCESSION NUMBER: 1989:614118 CAPLUS
DOCUMENT NUMBER: **111:214118**
TITLE: Process for the preparation of N,N-dimethyldiaminopropane
INVENTOR(S): Kiel, Wolfgang; Bauer, Wolfgang
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.
SOURCE: Eur. Pat. Appl., 3 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 316761	A2	19890524	EP 1988-118720	19881110
EP 316761	A3	19900704		
R: CH, DE, FR, GB, LI				
DE 3739260	A1	19890601	DE 1987-3739260	19871118

PRIORITY APPLN. INFO.: DE 1987-3739260 A 19871118

OTHER SOURCE(S): CASREACT 111:214118

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AB The efficiencies of three rigidly held cis-aquohydroxotetraazacobalt(III) complexes [(cyclen)**Co**(OH)(OH2)]2+ (cyclen = 1,4,7,10-tetraazacyclododecane), [(tren)**Co**(OH)(OH2)]2+ [tren = N(CH2CH2NH2)3], and [(trpn)**Co**(OH)(OH2)]2+ [trpn = N(CH2CH2CH2NH2)3] in promoting the hydrolysis of bis(p-nitrophenyl) phosphate (BNPP) have been compared. In neutral water at 50°, the rate constant for hydrolysis of the phosphate diester bond in [(cyclen)**Co**(OH)(BNPP)]+, [(tren)**Co**(OH)(BNPP)]+, [(trpn)**Co**(OH)(BNPP)]+ are 4.6 + 10-1, 8.1 + 10-3, and 2.5 s-1, resp. [(Trpn)**Co**(OH)(BNPP)]+ is hydrolyzed at about the same rate as BNPP bound to a real enzyme from Enterobacter aerogenes and about 1010 times more rapidly than free BNPP. The dramatic increase in the activity of the **Co**(III) complex with change in the tetraamine ligand structure can be explained in terms of a detailed mechanism of the reaction.

ACCESSION NUMBER: 1989:39085 CAPLUS
DOCUMENT NUMBER: **110:39085**
TITLE: **Cobalt**(III) complex-promoted hydrolysis of phosphate diesters: comparison in reactivity of rigid cis-diaquo(tetraaza)**cobalt**(III) complexes
AUTHOR(S): Chin, Jik; Banaszczyk, Mariusz; Jubian, Vrej; Zou, Xiang
CORPORATE SOURCE: Dep. Chem., McGill Univ., Montreal, QC, H3A 2K6, Can.
SOURCE: Journal of the American Chemical Society (1989), 111(1), 186-90
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 110:39085

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AB NC(CH2)nNR(CH2)mCN (R = H, CH2Ph, Me; m, n = 2,3), NCCH2CH2NH(CH2)4NHCH2CH2CN, and PhCH2NHCH2CH2CN were reduced by H in the presence of Raney **Ni** and NaOH to give H2N(CH2)n+1NR(CH2)m+1NH2. Debenzylation was avoided by the use of this catalyst.

ACCESSION NUMBER: 1985:95459 CAPLUS
DOCUMENT NUMBER: **102:95459**
TITLE: Amines and polyamines from nitriles
AUTHOR(S): Bergeron, Raymond J.; Garlich, Joseph R.
CORPORATE SOURCE: Dep. Med. Chem., Univ. Florida, Gainesville, FL, 32610, USA
SOURCE: Synthesis (1984), (9), 782-4
CODEN: SYNTBF; ISSN: 0039-7881
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 102:95459

L9 ANSWER 18 OF 20 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB By esterification of N,N'-bis(ω-hydroxyalkyl)piperazines and N,N'-bis(ω-hydroxyalkyl)-N,N'-dialkylpolymethylenediamines with 3,4,5-(MeO)3C6H2CO2H (I) and related alkoxybenzoic and alkoxyphenylacetic acids were prepared 48 sym. bis esters. Several of these open chain bis compds. showed a noteworthy pharmacodynamic action, particularly dilation of the coronary vessels. Multiple synthetic variations within this class of compds. demonstrated that the action was closely allied with entirely specific structural elements and that therefore a high specificity existed in the relation between constitution and action. (Distns. of smaller amts. were carried out by bulb to bulb distns.; b.ps. reported which range over 10° or more refer to such distns. and are air bath temps.) The required acid chlorides were prepared in the usual way with SOCl2. 3,4,5-(MeO)3C6H2CH2CO2H was prepared from 3,4,5-(MeO)3C6H2COCl (Ia) by Arndt-Eistert reaction. 3,4,5-(MeO)2[3,4,5-(MeO)3C6H2CO2]C6H2COCl (II), m. 138-43°, was prepared from the corresponding acid (IIa) and SOCl2 in 79% yield. Acylations with II in the presence of C5H5N resulted also in preparation of the anhydride of IIa, m. 245-8°, also prepared from II and the Et3N salt of IIa. Methods A. A mixture of 0.05 mole acid chloride (with higher melting acid chlorides the reaction was preferably carried out in absolute C6H6) and 0.055 mole appropriate halo alc. heated gradually to 100° (HCl evolved at 50-60° and the mixture became homogeneous), the product heated 3 hrs. on a water bath and diluted with Et2O, the solution washed with aqueous NaHCO3, dried, and evaporated, and the residue recrystd. from Et2O-petr. ether gave over 70% the following 3,4,5-RR1R2C6H2CO2(CH2)nY (III) (R, R1, R2, n, Y, m.p., b.p./mm. given): MeO, MeO, MeO, 2, Cl, 73-4°, -; H, MeO, H, 3, Cl, -, 100-10°/0.1; MeO, MeO, H, 3, Cl, 52-3°, -; MeO, H, MeO, 3, Cl, -, 160-70°/0.2; Cl, MeO, H, 3, Cl, -, 130-40°/0.4; Cl, MeO, Cl, 3, Cl, -, 110-20°/0.1; MeO, MeO, MeO, 3, Cl, 57-9°, -; MeO, MeO, MeO, 3, Br, 63-6°, -; MeO, MeO, MeO, 3, I, 53-7°, -; EtO, EtO, EtO, 3, Cl, 57-60°, -; MeO, MeO, MeO, 4, Cl, -, 160-70°/0.4. Also prepared were the following 3,4,5-RR1R2C6H2CH2CO2(CH2)nY (IV) (same data): H, MeO, H, 2, Cl, -, 90-5°/0.1; MeO, MeO, MeO, 2, Cl, 37-40°, -; H, MeO, H, 3, Cl, -, 105-10°/0.2; MeO MeO, MeO, 3, Cl, 18-20°, 165-70°/0.1. Likewise prepared was 85% 3,4,5-(MeO)3C6H2CO2CH2C.tplbond.CCH2Cl, m. 84-5° (prepared from Ia and ClCH2C.tplbond.CCH2OH in C6H6 containing absolute C5H5N at room temperature). (1) III or IV (preferably the bromo compound) (0.1 mole) and 0.1 mole anhydrous piperazine or N,N'-dialkylethylenediamine in 100 ml. dry HCONMe2 (DMF) heated 24 hrs. on a water bath, DMF evaporated at 10 mm., the residue partitioned between EtOAc and H2O, the organic phase separated, washed with H2O, and extracted with 2N HCl, the extract saturated with K2CO3 and extracted with Et2O (or C6H6 or EtOAc, according to the solubility of the product), and the extract dried and evaporated gave 40-80% bis ester, which was contaminated with difficulty separable monoester; the piperazine derivs. were recrystd. from MeOH-Et2O, with addition of petr. ether when necessary, and the noncrystg.

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dialkylethylenediamine derivs. were converted into salts. (2) The reaction was carried out without solvent by heating the components 20-4 hrs. at 100°, the product dissolved in EtOAc-Et2O, and the soln. filtered and worked up as in 1. (3) A soln. of 0.05 mole diamine in 50 ml. abs. DMF treated with 2.9 g. NaH, the mixt. heated 15 min. on a water bath, 0.1 mole III or IV added, the mixt. heated 2 hrs. at 100° and filtered, and the filtrate worked up as in 1 gave better yields of purer bis compds. Methods B. (1) Ethylene oxide (9.7 g.) introduced into 50 ml. MeOH contg. 8.8 g. (MeNHCH2)2 (V) with ice cooling, when the exothermic reaction subsided the cooling bath removed, and the soln. kept 1 hr. at room temp., heated 15 min. on a water bath, and fractionated gave 9.85 g. (HOCH2CH2NMeCH2)2, b0.04 105-7°, n20D 1.4828; dipicrate m. 220°. Similarly was prepd. 65% HO(CH2)2NMe(CH2)3NMe(CH2)2OH, b0.1 100-10°, n20D 1.4773; dipicrate m. 152-6°. (2) A hot (70-80°) soln. of 11.0 g. Na in 82 ml. CH2:CHCH2OH treated with 19.5 g. V, the mixt. heated 100 hrs. at 100-5° (bath), cooled, dild. with 100 ml. H2O, and extd. with Et2O, and the ext. fractionated gave 6.0 g. HO(CH2)3NMeCH2CH2NMe, b0.3-0.5 65-85° (dimethiodide m. 165-8°), and 17.1 g. [HO(CH2)3NMeCH2]2 (Va), b0.1 124-8° n20D 1.4830 (dimethiodide m. 200-5°; dipicrate m. 148-9°). (3) Piperazine or dialkylpolymethylenediamine (0.1 mole) treated gradually with 0.25 mole CH2:CHCO2Me (VI), when the exothermic reaction subsided the mixt. heated 1 hr. on a water bath, treated with 0.5 mole VI, heated 1 hr. more, and evapd. in vacuo, and the residue recrystd. (piperazine derivs.) from Et2O-petr. ether or distd. in vacuo gave over 65% following N,N'-bis(2-carbomethoxyethyl) derivs. (VII), (compd., b.p./mm., and m.p. dipicrate given): [MeO2C(CH2)2NRCH2]2 (VIII) (R = Me), 114-17°/0.3, 173-6°; VIII (R = Et), 125-7°/0.5, 144-8°; VIII (R = Pr), 120-5°/0.3, 153-7°; 1,4-bis(2-carbomethoxyethyl)piperazine, - (m. 53-5°), -; [MeO2C(CH2)2NEtCMe2]2 (VIIIa), 140-60°/0.3, -. The reaction with CH2:CMeCO2Me and V required more energetic conditions (20 hrs. on a water bath with a 5-molar excess of ester; addn. of some hydroquinone) and gave 20% (MeO2CCHMeCH2NMeCH2)2, b10 145-55° (dipicrate m. 167-71°), along with 30% MeO2CCHMeCH2NMeCH2CH2NHMe, b0.1 54-7° [dipicrate m. 66-70° (EtOH)]. Reaction of (H2NCH2)2 with 5 moles VI (18 hrs. on a water bath) gave 74% [(MeO2CCH2CH2)2NCH]2 (IX), b0.6 150-70°; dipicrate m. 164-7°. VII (0.1 mole) in 60-100 ml. abs. Et2O or tetrahydrofuran (THF) added dropwise to 5.0 g. LiAlH4 suspended in 50-70 ml. abs. Et2O (or THF) with stirring and cooling, the mixt. refluxed 8-10 hrs. (1 g. LiAlH4 was added after every 2 hrs.) and decompd. with the smallest possible amt. ice H2O, the ppt. filtered off and washed with Et2O, and the combined Et2O solns. evapd. gave over 60% bis propanol compds.; the compds. were recrystd. (piperazine derivs. from MeOH) or distd. in vacuo. Redn. of IX gave poorer yields (up to .apprx.30%) [(HOCH2CH2CH2)2NCH2]2 (IXa). (4) PhCH2NH(CH2)3OH (X) (28.2 g.) dissolved in 25 g. 80% HCO2H with cooling, 17 g. 35% aq. HCHO added, the soln. heated 16 hrs. on a water bath, cooled, treated with 20 ml. concd. HCl, and evapd. in vacuo, and the viscous residue made alk. with 40% aq. NaOH under Et2O and repeatedly extd. with Et2O gave 24.0 g. PhCH2NR(CH2)3OH (XI) (R = Me), b10 143-4°. X (69.8 g.) in 60 ml.

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EtOH treated portionwise with 140 g. EtBr with cooling and the soln. kept 3 days at room temp. in a closed vessel and fractionated gave 83 g. recovered EtBr, then EtOH, and finally 64.0 g. XI(R = Et), b12 148-50°,n25D 1.5165. XI(R = Me) (24 g.)in 150 ml. EtOH hydrogenated 6 hrs. over Raney Ni at 80-90° and 50 atm. H in a stirring autoclave gave 6.6 g. RNH(CH2)3OH (XII) (R = Me), b. 175-7° n20D 1.4479. Similar hydrogenation of XI (R = Et) (100°, 100 atm. H, 12 hrs.) gave 85% XII (R = Et), b. 184-7°, n22 1.4475. CH2:CHCO2Et (22 g.) added dropwise during 60 min. to 10 g. MeNH2 in 60 ml. EtOH at 40-50° with stirring and the mixt. heated 1 hr. on a water bath gave 14.5 g. MeNH(CH2)2CO2Et, b12 60-2° (dipicrate m. 90-2°), which reduced with LiAlH4 in Et2O gave 68% XII (R = Me). XII (R = Me) (8.2 g.) heated to 120-30° with stirring, 7.0 g. (CH2Br)2 added dropwise during 25 min., and the mixt. heated and stirred 2 hrs. at 120°, cooled, dissolved in 10 ml. hot H2O, treated with 5 ml. 40% aq. NaOH, and continuously extd. with Et2O gave 3.4 g. [HO(CH2)3NRCH2]2 (XIII) (R = Me). Similarly was prepd. from XII (R = Et) [3 moles XII (R = Et)/ mole (CH2Br)2 used; heated 5 hrs. at 120°] 64% XIII (R = Et) along with 30-40% HO(CH2)3NEtCH2CH2NHEt. (CH2Cl)2 (50 g.) added dropwise during 80 min. to 150 g. H2N(CH2)3OH (XIV) heated to 110° (bath) with stirring, the mixt. stirred 4 hrs. at 110°, cooled to 40-50°, and dissolved in 500 ml. EtOH, 40 g. finely powd. NaOH added with stirring, after a time the pptd. NaCl filtered off and washed twice with 50 ml. EtOH and twice with 50 ml. MeOH, and the combined filtrates neutralized with concd. HCl, filtered, and fractionated gave 84.5 g. recovered XIV, b12 81-5°, and 53.6 g. [HO(CH2)3NHCH2]2 (XV), b0.1 155-60°, which solidified during distn. (m. .apprx.70°) and necessitated flushing the condenser with hot water [XV.2HCl m. 152-5° (EtOH); XV dipicrate m. 214-16°]; XV contained 4-5% N,N'-bis(3-hydroxypropyl)piperazine (XVI) which crystd. from MeOH at 0°. XV (39.3 g.) dissolved in 62 g. 85% HCO2H with cooling, the soln. heated on a water bath, 50 g. 35% aq. HCHO added dropwise with stirring, the soln. heated 24 hrs. and evapd. in vacuo, and the residue evapd. with H2O and EtOH, treated with 30-40 ml. 40% aq. NaOH (pH 8-9), heated 1 hr. on a water bath, and continuously extd. with Et2O (24 hrs.) gave after distn. 24.8 g. Va; Va contained 2-3% XVI. Va (25 g.) in 100 ml. Et2O treated with 40 ml. petr. ether pptd. 0.5-0.75 g. XVI, m. 135-40°. X (30.0 g.) treated dropwise at 110° with 14.0 g. (CH2Br)2 with stirring, the mixt. heated and stirred 3 hrs. at 100-10° and made alk. with concd. aq. NaOH, and the product isolated by repeated extn. with C6H6 gave 8.8 g. unchanged X, b0.4 89-101°, and then 18.5 g. [HO(CH2)3N(CH2Ph)CH2]2, b0.04 214-18°, n20D 1.5510 [dipicrate m. 200-3° (Me2CO-MeOH)]. (5) Anhyd. piperazine (8.6 g.) in 80 ml. C6H6 treated portionwise with 15.9 g. MeO2CCH2CH2COCl in 30 ml. C6H6, the mixt. kept 4 hrs. at room temp. and filtered hot, the filter cake washed repeatedly with hot C6H6, and the combined filtrates evapd. in vacuo gave 14.0 g. N,N'-bis(β-carbomethoxypropionyl)piperazine, m. 122-4° (C6H6-Et2O). Similarly were prepd. 80% N,N'-dimethyl-N,N'-bis(β-carbomethoxypropionyl)ethylenediamine (XVII), b0.05 190-200°, 82% N,N'-di-Et analog of XVII, m. 66-70°, and 76% N,N'-bis(γ-carbomethoxybutyryl)piperazine, m. 60-3° (C6H6-Et2O). Redn. of these compds. with LiAlH4 in THF (boiling 18 hrs.) gave the corresponding bis alkanols. 3,4,5-(MeO)3C6H2CO2(CH2)4Cl and V in DMF (or PhMe) heated several hrs., the soln. evapd. in vacuo, the residue partitioned between EtOAc and H2O, and the aq. phase acidified gave I; the EtOAc washed with

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dil. HCl and evapd. and the residue repeatedly recrystd. from MeOH gave .apprx.30% 3,4,5-(MeO)3C6H2CO2(CH2)nO2CC6H2(OMe)3-3,4,5 (XVIII) (n = 4), m. 118-22°, also obtained by reaction of HO(CH2)4OH and Ia in abs. dioxane in the presence of C5H5N; the acid soln., according to its chromatogram, contained a mixt. of 5 amines. From HO(CH2)3OH and Ia was similarly obtained XVIII (n = 3), m. 90-2° (MeOH-Et2O). [Me2C(NH2)]2 heated 45 min. on a water bath with 2.2 moles Ac2O in Et2O gave 86% N,N'-di-Ac deriv., m. 168-9°, which boiled 16 hrs. in THF with excess (4 moles) LiAlH4 gave 60% [Me2C(NHEt)]2, b10 71-4° [dipicrate m. 192-6° (EtOH-H2O)], which treated with VI (large excess, 15 hrs. on a water bath) gave VIIIa, reduced to the bis propanol (XVIIIa) with LiAlH4 in Et2O or THF. Redn. of (MeO2CCH2CH2)2NEt with LiAlH4 in Et2O gave 60% [HO(CH2)3]2NEt (XIX), b0.2 107-9°, n20D 1.4702. The phys. consts. of the following addnl. bis alkanols were recorded (compd., method of synthesis, b.p./mm., m.p., n20D, m.p. dipicrate given): XIII (R = Et), B-2 and B-3, 125-30°/0.5, -, -, 151-2°; XIII (R = Pr), B-3, 140-50°/0.3, -, 1.4752, 153-6°; HO(CH2)3NMe(CH2)3NMe(CH2)3OH, B-3, 120-30°/0.2, -, -, -,; [HO(CH2)4NRCH2]2, (XX) (R = Me), B-4, 160-70°/0.5, -, 148-52°; XX (R = Et), B-4, 130-40°/0.2, -, 1.4772, 114-17°; IXa, B-3, 170-95°/0.1, -, -, 190-5°; XVI, B-3, -, 142-4°, -, -; N,N'-bis(4-hydroxybutyl)piperazine, B-4, -, 114-16°, -, -; N,N'-bis(5-hydroxypentyl)piperazine, B-4, -, 110-12°, -, -; XVIIIa, B-3, 160-80°/0.2, 88-93°, -, -; (HOCH2CHMeCH2NMeCH2)2, B-3, 100-10°/0.1, -, -, 149-52°. To 8.0 g. XIX and 18 g. C5H5N in 80 ml. abs. dioxane was added dropwise during 30 min. 24.2 g. Ia in 60 ml. abs. dioxane with stirring at room temp., the mixt. heated and stirred 3 hrs. at 100° and evapd. in vacuo, the residue dissolved in EtOAc, the soln. dried and evapd., the residue dissolved in Et2O, some petr. ether added, the soln. cooled in ice overnight, the ppt. [1.4 g. (3,4,5-(MeO)3C6H2CO]2O] filtered, and the filtrate evapd. gave 23.6 g. [3,4,5-(MeO)3C6H2CO2(CH2)3]2NEt; HCl salt m. 170-5° (90% EtOH). Reaction of appropriate acid chloride with bisalkanols to bisesters was effected either (as described above) in abs. dioxane (or C6H6) with addn. of C5H5N (or another tertiary amine) or without addn. of base. The yields were between 60-90%. When base was not added, di-HCl salts of bisesters sepd. after some time. The mixt. was heated 3 hrs., dild. with Et2O, and satd. with HCl to complete the pptn. of the HCl salts. When the HCl salts did not sep. as solids, the mixt. was evapd., the residue treated with concd. aq. Na2CO3, and the product extd. with Et2O or EtOAc and converted to the HBr salt. The synthesis of the better crystg. piperazine derivs. was advantageously carried out by treating the bisalkanol with 2.1 moles acid chloride in boiling PhMe (or abs. dioxane). Satn. with HCl gave the di-HCl salt in over 60% yield. The bis(4-aminobenzoic acid esters) and bis(4-acetamidobenzoic acid esters) were obtained by hydrogenation of the corresponding NO2 compd. in EtOH over Raney Ni followed by acetylation with Ac2O. The following bisesters, 3,4,5-RR1R2C6H2CO2(CH2)n NZ(CH2)m NZ(CH2)nO2CC6H2RR1R2-3,4,5, were prepd. (method, Z, m, n, R, R1, R2, salt, m.p. salt given): by debenzylation of the corresponding PhCH2 deriv., H, 2, 3, MeO, MeO, MeO, di-HCl, 187-93°; A, Me, 2, 2, MeO, MeO, MeO, di-HCl, 173-6°; B, Me, 3, 2, H, NO2, H (XXI), -, 200-4° (decompn.) (free base); by hydrogenation of XXI, Me, 3, 2, H, NH2, H, di-HCl, above 130° (decompn.) [bis (H oxalate) m. 193-5° (decompn.)]; B, Me, 2, 3, H, H, H, di-HCl, 188-93°; B, Me, 2, 3, H, MeO, H, di-HCl, 184-9°; B, Me, 2, 3, MeO, MeO, H, di-HCl, 228-31°; B, Me, 2, 3, MeO, H, MeO, di-HCl, 134-9°; B, Me,

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2, 3, Cl, MeO, H, di-HCl, 168-73°; A and B, Me, 2, 3, MeO, MeO, MeO (XXII), di-HCl, 170-4° (free base m. 75-7°; di-HBr salt m. 196-200°); B, Me, 2, 3, EtO, EtO, EtO, di-HBr.2H2O, 63-8°; B, Me, 2, 3, MeO, 3,4,5-(MeO)3C6H2CO2, MeO, di-HBr, 166-75°; B, Me, 2, 3, H, NO2, H, di-HCl, 204-10°; B, Me, 2, 3, H, NHAc, H, di-HCl, 149-52°; A and B, Me, 3, 3, MeO, MeO, MeO, di-HCl, 156-9°; A, Me, 4, 3, MeO, MeO, MeO, di-HCl, 193-7°; A, Me, 10, 3, MeO, MeO, MeO, di-HCl, 70-85°; B, Me, 2, 4, MeO, MeO, MeO, di-HCl, 152-7°; B, Me, 2, [(CH2)n =] CH2CHMeCH2, MeO, MeO, MeO, di-HCl, 200-3°; A and B, Et, 2, 3, MeO, MeO, MeO (XXIII), di-HBr.H2O, 76-9°; B, Et, 2, 3, EtO, EtO, EtO, di-HBr. H2O, 53-60°; B, Et, 2, 3, MeO, 3,4,5-(MeO)3C6H2CO2, MeO, di-HBr, 170-8°; B, Et, [N(CH2)n N =] NCMe2CMe2N, 3, MeO, MeO, MeO, monopicrate, 140-5°; B, Et, 2, 4, MeO, MeO, MeO, di-HBr, 182-6°; B, Pr, 2, 3, MeO, MeO, MeO, di-HBr, 200-5°; B, Pr, 2, 3, EtO, EtO, EtO, di-HBr, 167-74°; B, PhCH2, 2, 3, MeO, MeO, MeO, di-HCl, 97-100°; B, 3,4,5-(MeO)3C6H2CO2(CH2)3, 2, 3, MeO, MeO, MeO, di-HCl, foam. Similarly prepd. were the following 3,4,5 - RR1R2C6H2CH2CO2(CH2)nNZ(CH2)nNZ(CH2) O2CCH2C6H2RR2-R-3,4,5: A, Me, 2, 2, MeO, MeO, MeO, di-HCl, 152-60°; A and B, Me, 2, 3, H, MeO, H, di-HCl, 170-5°; B, Me, 2, 3, MeO, MeO, MeO, di-HCl, 121-7°; B, Me, 2, 4, H, MeO, H, di-HCl, 156-61°. The following piperazine derivs. (XXIV) were prepd.: (method, n, R, R1, R2, R3, m.p., salt, m.p. salt given): B, 2, H, H, MeO, H, 98-101°; di-HCl, 235-40°; B, 2, MeO, MeO, H, H, -, di-HCl, 198-204°; B, 2, H, MeO, H, MeO, -, di-HCl, 200-5°; B, 2, H, MeO, MeO, MeO, -, di-HCl, 215-18°; B, 2, H, Cl, MeO, Cl, 136-9°, di-HCl, 202-5°; A and B, 3, H, MeO, MeO, MeO (XXV), 116-19°, di-HCl, 216-20° (di-HBr salt m. 222-7°); A and B, 3, H, H, NO2, H (XXVI), -, di-HCl, 218-22°; by hydrogenation of XXVI, 3, H, H, NHAc, H, 187-90°, di-HCl, 228-33°; B, 4, H, H, MeO, H, 130-2°, di-HCl, 217-23°; B, 4, H, MeO, MeO, H, 97-100°, di-HCl, 202-6°; B, 4, H, Cl, Cl, H, 97-9°, di-HCl, 210-12°; B, 4, H, MeO, MeO, MeO, 100-4°, di-HCl, 195-200°; B, 4, H, Cl, MeO, Cl, 111-15 , di-HCl, 189-91°; B, 4, H, EtO, EtO, EtO, 88-90°, di-HCl, 197-205°; B, [(CH2)n =] CH2C.tplbond.CCH2, H, MeO, MeO, MeO, 157-9°, di-HCl, 185-93°; B, 5, H, MeO, MeO, MeO, 91-3°, di-HCl, above 200° (decompn.). Pharmacol. and clinical investigation indicate that many of the trimethoxybenzoic ester compds. (particularly XXII, XXIII, and XXV) possess, along with the expected local anesthetic and antihistaminic activity, antifibrillatory and coronary vessel dilating activity, so that they appear suitable for various cardiac diseases.

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GI For diagram(s), see printed CA Issue.
AB cf. C.A. 52, 2859f. N-(5-Bromophenyl)phthalimide (220 g.), 480 g. EtNH(CH2)2OH, and 2 l. xylene refluxed 18 hrs., cooled, treated with 1 mole K2CO3, and evaporated in vacuo, the residue extracted with CH2Cl2, the extract evaporated, the residual crude N-[5-[ethyl(2-hydroxyethyl)amino]pentyl]phthalimide treated with 200 cc. concentrated HCl and 200 cc. H2O with cooling, the mixture refluxed 4 hrs., cooled, and filtered, the residue washed with 25 cc. H2O, the combined filtrates basified strongly with saturated aqueous KOH and saturated with solid KOH, and the oily layer dried with solid KOH and distilled gave 45 g. H2N(CH2)5NEt(CH2)2OH, b1 103-4°, n25D 1.4870. AmNH(CH2)2OH (52.6 g.) treated below 30° with stirring during 7 min. with 23.4 g. CH2:CHCN (I), stirred 2 hrs. at room temperature, heated 1 hr. at 80°, kept 18 hrs. at room temperature, and stirred 2 hrs. in vacuo, the residue (72 g.) dissolved in 300 cc. EtOH (saturated with NH3), hydrogenated 1.5 hrs. at 100° and 1100 lb. over Raney Ni, and filtered, and the filtrate distilled gave 54.5 g. H2N(CH2)3NAm(CH2)2OH, b16 162-5°, n28D 1.4664. I (106 g.) added dropwise with stirring to 200 g. 3-piperidinol at 30-40° with cooling, kept 18 hrs. at room temperature, and heated 2 hrs. on the steam bath (the last hr. in vacuo) gave crude 3-hydroxy-1-piperidinepropionitrile (II). The crude II and 90 cc. Et3N hydrogenated 40 min. at 70-102° and 1775 lb. over 90 g. moist Raney Co (previously washed with absolute EtOH and cyclohexane), diluted with EtOH and cyclohexane, and evaporated in vacuo on the steam bath, and the residue distilled gave 234 g. 1-(3-aminopropyl)-3-piperidinol, b0.07 79-80°, n25D 1.5022. 7-Chlorobenz[c]acridine (III) (0.038-0.080 mole), 0.042-0.085 mole of the appropriate aminoalkylaminoalkanol, and 40-60 g. PhOH heated 3-4 hrs. on the steam bath, cooled, poured into 10-20 cc. concentrated HCl in 125-200 cc. Me3CO, cooled, and diluted with 200-400 cc. Me2CO or Et2O gave the corresponding [(benz[c]acridin-7-ylamino)alkylamino]alkanol-2HCl (IV.2HCl) (method A). III (0.030-0.038 mole) and 25-40 g. PhOH heated with stirring to 120°, cooled to 80°, treated with 0.035-0.042 mole of the appropriate diamine, heated 3 hrs. with stirring at 80-110°, poured into a solution of 5-90 g. KOH in 300-400 cc. H2O, and extracted with Et2O or CHCl3, and the extract dried, decolorized, and treated with dry HCl or an appropriate acid in Et2O gave the corresponding IV salt (method B). In this manner were prepared the following V(X, R, salt-forming acid, moles H2O of crystallization, m.p. of salt, % yield, and method given): (CH2)2, H, 2HCl, 0, 147° (decomposition) (MeOH-Et2O), 38, A; CH2CH(Me), H, 2HCl, 2, indefinite at 85° (EtOH-EtOAc), 80, A; (CH2)4 3, H, 2HCl, 1, 215-20°, -, -; (CH2)3, Et (VI), 2HCl, O, 223-5° (decomposition) (MeOH-Me2CO), 52, A (57% by method B); (CH2)2, Am, 2HCl, 2.25, 150° (EtOH-EtOAc), 60, A; (CH2)5, Et, 2o-HOC6H4CO2H, 0.25, 133-5° (decomposition) (MeOH), 63, B; (CH2)2, (CH2)2OH (VII), 2HCl, O, 220-30° (MeOH), 91, A. III (0.030 mole) and 26 g. PhOH treated slowly at 80° with 0.035 mole H2N(CH2)3N(CH2CH2OH)2, heated 3 hrs.

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with stirring at 100°, cooled, and slowly poured into 90 g. KOH in 300 cc. H2O, the aq. layer decanted, the gummy residue extd. with dil. HCl, the acidic ext. basified and filtered, and the residue treated with Me2CO and C and then with dry HCl yielded 43% VII. III (0.100 mole), 0.105 mole MeCH(NH2)(CH2)3NEtCH2CH2OH, and 60 g. PhOH heated 2 hrs. with stirring at 120-30°, cooled, stirred into 20% aq. NaOH, and extd. with CHCl3, the ext. worked up, the residual oil dissolved in EtOH, acidified with alc. HCl, dild. with Me2CO-Et2O to give a dark tarry ppt., the solvents evapd., the residue basified with NH4OH and extd. with Et2O, the ext. washed and extd. with Et2O, the ext. washed, dried, and treated with alc. HCl, and the orange ppt. dissolved in MeOH, evapd., powdered, and dried 24 hrs. at room temp. yielded 45% V [X = CHMe(CH2)3, R = Et].2HCl.0.5H2O, m. 135-40°. III (55.5 g.), 40 g. MeCH(NH2)(CH2)3N(CH2CH2OH)2, and 250 g. PhOH heated 4 hrs. with stirring on the steam bath, cooled, and poured into 2 l. Me2CO contg. excess concd. HCl, the pptd. orange-red tar triturated with Me2CO and Et2O, the crude solid recrystd. twice from EtOH, the resulting dull yellow solid dissolved in H2O, the soln. poured into excess NH4OH and extd. with CHCl3, the ext. dried, concd. in vacuo to 100 cc., dild. with 1.5 l. Et2O, treated with dry HCl, and filtered, and the residue equilibrated in air gave 37% V [X = CHMe(CH2)3, R = CH2CH2OH], m. 80° (indefinite) (EtOH). V [X = (CH2)3, R = Et].2HCl.H2O (40 g.) in H2O treated with C, filtered, and basified with NH4OH, the aq. soln. decanted, and the residue treated with Et2O yielded 28 g. VI, m. 80-2° (decompn.) (MeOH). By method B was prepd. in the usual manner 3-(benz[c]acridin-7-ylamino)propylaminopropanol, m. 70° (indefinite). III (20 g.), 12.7 g. Et2NCH2CH(OH)CH2NH2, and 75 g. PhOH heated 2 hrs. with stirring at 110°, kept 16 hrs. at room temp., poured with stirring into 175 g. KOH in 1 l. H2O contg. 500 g. ice, and extd. with Et2O, the ext. washed, decolorized, dried, and treated with dry HCl, and the ppt. washed with Et2O and Me2CO and dried 24 hrs. at 40° in vacuo gave 14.2 g. (benz[c]acridin-7-ylamino)-3-diethylamino-2-propanol, hygroscopic yellow powder, m. 100° (indefinite). III (26.3 g.), 16.6 g. II, and 50 g. PhOH heated 3 hrs. with stirring on the steam bath, cooled, treated with excess alc. HCl, dild. with 1 l. dry Me2CO, chilled, and filtered, the residue washed with dry Me2CO, added to excess NH4OH, and extd. with CHCl3, the ext. washed, decolorized, and evapd. in vacuo, the oily residue dissolved in abs. EtOH, treated with excess alc. HCl, stirred and warmed with MeOH, dild. with dry Me2CO, chilled, and filtered yielded 42.5 g. 1-[3-(benz[c]acridin-7-ylamino)propyl]-3-piperidinol-2HCl.H2O, m. 262°. The appropriate IV.2HCl (0.009-0.076 mole) dried in vacuo 18-48 hrs. at 35-100°, stirred and heated 7-24 hrs. on the steam bath in 40-500 cc. of the appropriate acid chloride, kept 1-16 hrs. at room temp., dild. with Et2O, and filtered, and the residue dried in vacuo at room temp. 5-18 hrs. and recrystd. gave the corresponding esters of IV (method C). IV.2HCl (0.011-0.034 mole) dried 18 hrs. at 35-100°, heated 20-4 hrs. with 0.011-0.040 mole succinic anhydride at 100-50° under N, the mixt. dissolved in boiling abs. EtOH, the soln. dild. with dry Et2O, and the yellow ppt. filtered off, washed with Et2O, and dried gave the ester of IV (method D). In this manner were prepd. the following esters of V.2HCl (X, R, ester, moles H2O of crystn., m.p., % yield, and method given): (CH2)2, H, acetate, 0.5, 135-45°, 100, C; (CH2)2, H, palmitate, 1, 170-80° (MeOH), 67, C; (CH2)2, H, 0.5, H succinate, 80° (decompn.), 77, D; (CH2)3,

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Et, acetate, 1, 174-6°, 93, C; (CH2)3, Et, heptanoate, 2, 147-9°, 80, C; (CH2)3, Et, palmitate, 3, 168-71°, 92, C; (CH2)3, Et, H succinate, 1, 120° (HCONMe2-Et2O), D (under N); (CH2)3, (CH2)2OH, H succinate, 0.25, 80° (decompn.), 90, D; (CH2)3, (CH2)2OCOC15H33, palmitate, 2.5, 185-6° (MeOH), 85, C. III (10 g.) and 40 g. PhOH heated 15 min. with stirring on the steam bath, heated 2 hrs. with stirring with 6.1 g. H2N(CH2)3NH(CH2)2OH on the steam bath, poured into 500 cc. 10% aq. NaOH, and extd. with Et2O, the ext. washed, dried, and treated dropwise with 2.9 g. (CH2COCl)2 with shaking, and the mixt. kept 1 hr. at room temp., treated with dry HCl, and filtered gave 16 g. di-2-{[3-(benz[c]acridin-7-ylamino)propyl]ethylamino}ethyl succinate-HCl.3H2O, m. 130-5° with softening at 80°.

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TITLE: Synthetic amebicides. IV. [(Benz[c]acridin-7-ylamino)alkylamino]alkanols and their esters
AUTHOR(S): Elslager, Edward F.; Short, Franklin W.; Sullivan, Marie Jo; Tendick, Frank H.
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AB The basically substituted propionitriles are best obtained by the addition of NH3 or primary or secondary amines to CH2:CHCN (I); γ-aminobutyronitriles were prepared from Cl(CH2)4NH2 and secondary amines; the yields are greatly improved by the use of a solvent. The mechanism by which the addition takes place probably involves a typical 1,4-addition Thus β-(3-diethylaminopropylamino)propionitrile (b25 163-5°, n20D 1.4573; picrate, m. 123°) results in 79.4% yield from I and H2N(CH2)3NEt2 and in 76.4% yield from Br(CH2)2CN and H2N(CH2)3NEt2. The reaction is reversible. When the higher β-dialkylaminopropionitriles are heated near their b. p. for any length of time, they slowly decompose to give some of the R2NH. (HOCH2CH2)2NH (II) and I, allowed to stand 8 h. at room temperature, give (HOCH2CH2)2NCH2CH2CN (III), which could be isolated as the picrate, m. 108-9°; however, attempted distillation gave 67% of III and a nondistillable polymer. Reduction of III in EtOH at 105-10° and 133 atmospheric gives 40% of 3-diethanolaminopropylamine, b2 158°, n 1.4975 (all n are n20D) (picrate, m. 156.5-7.5°). H2NCH2CH2CN (IV) (b5 66-9°, n 1.4396; picrate, m. 178°), on standing, gives NH3 and a polymer, best explained by its disproportionation into NH3 and I. The rate of addition of amines to I is discussed; this is not a function of the basic strength of the amines but is more dependent upon the size and shape of the entering mol. In certain cases a catalyst is necessary to induce reaction. In the reaction of I with NH3, NH(CH2CH2CN)2 (V) (b4 165°, n 1.4640) and N(CH2CH2CN)3 (VI) predominate when NH4OH is used but with a 7-mol. excess of liquid NH3 at 40°, the products are 22% of IV and 64% of V, with very little VI. Thus V adds to I approx. 20 times faster than NH3. I (424 g.) and 1000 g. EtNH2, warmed for 24 h. at 50° and allowed to stand 2 days at room temperature give 97% of Et2NCH2CH2CN (VII), b735 196°, n 1.4356; picrate, m. 85°; by using mol. ratios and heating on the steam bath for 8 h., the yield is 74%; 5 mols. of EtNH2 and 9.5 mols. of com. I, refluxed 0.5 h. and kept at 0° overnight, give 93% of VII. In general, better yields result if the reaction product is allowed to reach equilibrium at room temperature before distillation; the product should be distilled more or less rapidly at a low temperature if possible. β-Ethylaminopropionitrile, b30 92°, n 1.4318, 90.4%; picrate, m. 163°. Bis(2-cyanoethyl)ethylamine, b30 200-2°, n 1.4591, 60%; picrate, m. 170°. Pr2NCH2CH2CN, b20 116°, n 1.4381, 88%; picrate, m. 111°. Bu2NCH2CH2CN, b20 141°, n 1.4423, 91%; picrate, m. 75°. β-Diamylaminopropionitrile, b19 159-61°, n 1.4457, 89%; the picrate is an oil. β-Dihexylaminopropionitrile, b2 145-6°, n 1.4483, 85%. β-(1-Piperidyl)-2-propionitrile, b30 129-30°, n 1.4697, 93°; picrate, m. 160°. β-(4-Morpholinyl)propionitrile, b20 149°, n 1.4710, 95%; picrate, m. 139.5°. Bis(2-cyanoethyl)ethanolamine picrate, m. 137-8°. 1-Diethylamino-3-[bis(2-cyanoethyl)amino]propane, b25 233-5°, n 1.4709, 8.8%; picrate, m. 166-7°. I and (Et2NCH2CH2CH2)2NH with 0.1 g. Cu bronze, heated at 100° for 24 h. and allowed to stand 24 h. at room temperature, give 78% of β-[bis(3-diethylaminopropyl)amino]propionitrile, b3 153°, n 1.4640; picrate, m. 157-8°. β-[2-(4-Morpholinyl)ethylamino]propionitrile, b20 183°, n 1.4817, 81.5%; picrate, m. 176.5°; β-[3-(4-morpholinyl)propylamino]propionitrile, b9 178-80°, n 1.4819, 76%; picrate, m. 148-9°. β-(2-Diethylaminoethoxy)propionitrile, prepd. in 80% yield by adding 100 g. I to 220 g. Et2NCH2CH2OH and 2.3 g. MeONa during 0.5 h. at 25°, allowing to stand overnight, treating with 4.2 mL. concd. H2SO4 and distg. the filtrate, b25 145°, n 1.4430; picrate, m. 75°; β-(3-diethylaminopropoxy)propionitrile, b25 148-50°, n 1.4440, 75.4%. β-(4-Diethylamino-1-methylbutoxy)propionitrile, b3 125-30°, n 1.4456, 66%. I (27 g.) and 53 g. PhNHMe do not react when heated at 180° for 4 h.; in the presence of 1 g. CuSO4.5H2O there results 20 g. of β-(methylphenylamino)propionitrile, b20 175-7°; no reaction occurs with PhCH2NMe3OH in dioxane at 100°; picrate, m. 118°. I (250 mL.) and 167 g. carbazole, cooled in an ice-bath and treated with 2 cc. 40% PhCH2NMe3OH, with heating on the steam bath for 1 h., give 85.4% of 9-(2-cyanoethyl)carbazole, m. 155.5°. I and tetrahydroquinoline did not react with the usual catalysts at 160° after 4 h.; addn. of 100 g. I to 133 g. of tetrahydroquinoline at 125°, with refluxing for 6 h., gives 75.5% of 1-(2-cyanoethyl)tetrahydroquinoline, b10 192°, n 1.5780; picrate, m. 172°. γ-Diethylaminobutyronitrile, b21 101-3°, n 1.4351, 86%; picrate, m. 69-70°; γ-(1-piperidyl)butyronitrile, b25 127-9°, n 1.4653, 87%; picrate, m. 117°; γ-(4-morpholinyl)butyronitrile, b25 148-50°, n 1.4665, 70%; picrate, m. 152-3°. The redn. of the nitriles to amines was carried out with approx. 10 g. Raney Ni per mol. of nitrile at temps. of 90-130° and H pressures of 67 to 270 atm. The formation of secondary amines (5-32%) was obsd., being greater in the case of the γ-aminobutyronitriles; the redn. can be controlled and the yield of primary amine increased by carrying out the hydrogenation in the presence of NH3; the yield of secondary amines can be raised by reducing the nitrile in the presence of an excess of the primary amine. It is believed that the yields of primary products can be increased in almost every case if H pressures of 250-300 atm. are employed and larger amts. of NH3 are introduced. The original gives the pressure, temp., time and solvent used for the various redns. The following compds. are reported. (CH2)3(NH2)2, b735 138°, n 1.4600, 23%; picrate, m. 178°. Et2N(CH2)3NH2, b735 168°, n 1.4355 (picrate, m. 194°), yields up to 72%; bis(3-diethylaminopropyl)amine, b3 107°, n 1.4541 (picrate, m. 153-4°), yields up to 29%. 3-Ethylaminopropylamine, b735 156°, n 1.4441, 74%; picrate, m. 193°. Bis(3-aminopropyl)ethylamine, b20 135°, n 1.4709, 16%; picrate, m. 197-9°. Pr2N(CH2)3NH2, b20 94°, n 1.4435, 49%; picrate, m. 181°; Bu2N(CH2)3NH2, b20 121°, n 1.4462, 32%; picrate, m. 188°. 3-(1-Piperidyl)propylamine, b730 205°, n 1.4750, 68.5%; picrate, m. 209-10°. Bis[3-(1-piperidyl)propyl]amine, b2 153°, n 1.4916, 10%; picrate, m. 193°. 3-(4-Morpholinyl)propylamine, b733 219°, n 1.4762, 70.6%; picrate, m. 166°. Bis[3-(4-morpholinyl)propyl]amine, b5 185°, n 1.4918, 10%; picrate, m. 213-15°; in the prepn. of the primary amine, up to 35% morpholine are formed under certain conditions. 4-Et2N(CH2)4NH2, b18 85-8°, n 1.4462, 51%; picrate, m. 155-6°. 4-(1-Piperidyl)butylamine, b25 118-20°, n 1.4756, 53.8%; picrate, m. 160.5°. Bis[4-(1-piperidyl)butyl]amine, b25 220-5°, n 1.4898, 32%; picrate, m. 202-3°. 4-(4-Morpholinyl)butylamine, b20 122°, n 1.4760, 62%; picrate, m. 148°. Bis[4-(4-morpholinyl)butyl]amine, b3 200-2°, n 1.4900, 23.8%; picrate, m. 136°. Bis(3-aminopropyl) ether, b32 113°, n 1.4618, 29%. 3-(3-Diethylaminopropylamino)propylamine, b25 142-4°, n 1.4630, 51%; picrate, m. 197-8°; bis[3-(3-diethylaminopropylamino)propyl]amine, b25 253-60°, n 1.4710, 31%; picrate, m. 197°. 3-[Bis(3-diethylaminopropyl)amino]propylamine b3 155-65°, n 1.4662, 52%; picrate, m. 162.5°. 3-[2-(4-Morpholinyl)ethylamino]propylamine, b2 120-3°, n 1.4870, 57.5%; picrate, m. 208°. 3-[3-(4-Morpholinyl)propylamino]propylamine, b1.5 137-40°, n 1.4878, 45.2%; picrate, m. 205°. 3-(2-Diethylaminoethoxy)propylamine, b25 118-22°, n 1.4498, 56.7°. Bis[3-(2-diethylaminoethoxy)propyl]amine, b3 175°, n 1.4582, 23.8%. 3-(3-Diethylaminopropoxy)propylamine, b25 130-2°, n 1.4500, 57.4%. Bis[3-(3-diethylaminopropoxy)propyl]amine, b3 182°, n 1.4581, 28.2%. 3-(4-Diethylamino-1-methylbutoxy)propylamine, b2 80-3°, n 1.4492, 50.5%; picrate, m. 88-9°. Bis[3-(4-diethylamino-1-methylbutyoxy)propyl]amine, b3 210-15°, n 1.4580, 23%. 3-(Methylphenylamino)propylamine, b40 171-2°, 63%; HBr salt, m. 120°; picrate, m. 189°. 9-(3-Aminopropyl)carbazole, b3 228°, 70.5%; HCl salt, m. 273°; picrate, m. 206-7°. 1-(3-Aminopropylamino)tetrahydroquinoline, b3 132-5°, n 1.5828, 82%. The following N1-sulfanilamide derivs. were prepd. through the N4-Ac derivs.; in some cases the Ac derivs. were viscous sirups which did not crystallize. 3-Diethylaminopropyl, m. 109-10°, 20%; 3-dipropylaminopropyl, m. 98-8.5°, 57°; 3-dibutylaminopropyl, HCl salt, m. 110-15°, 53.5%; 3-(1-piperidyl)propyl, m. 105.5-6°, 63.5% (Ac deriv., m. 109-11°); 3-(4-morpholinyl)propyl, m. 94.5-5°, 79% (Ac deriv., m. 97-8°); bis(3-diethylaminopropyl), HCl salt, m. 195-7°, 66.5% (Ac deriv., m. 83-5°); bis[3-(1-piperidyl)propyl], m. 74-6°, 71%. It was not possible to use C5H5N or C5H5N-Me2CO as solvents in the prepn. of these derivs.

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ORIGINAL REFERENCE NO.:	38:3617b-i,3618a-i,3619a-c
TITLE:	Basically substituted aliphatic nitriles and their catalytic reduction to amines
AUTHOR(S):	Whitmore, Frank C.; Mosher, Harry S.; Adams, Robert R.; Taylor, Robert B.; Chapin, Earl C.; Weisel, Charles; Yanko, Wm.
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ANSWER 1 CASREACT COPYRIGHT 2005 ACS on STN
AN 38:24921 CASREACT
TI Basically substituted aliphatic nitriles and their catalytic reduction to amines
AU Whitmore, Frank C.; Mosher, Harry S.; Adams, Robert R.; Taylor, Robert B.; Chapin, Earl C.; Weisel, Charles; Yanko, Wm.
SO Journal of the American Chemical Society (1944), 66, 725-31
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AB The basically substituted propionitriles are best obtained by the addition of NH3 or primary or secondary amines to CH2:CHCN (I); γ-aminobutyronitriles were prepared from Cl(CH2)4NH2 and secondary amines; the yields are greatly improved by the use of a solvent. The mechanism by which the addition takes place probably involves a typical 1,4-addition Thus β-(3-diethylaminopropylamino)propionitrile (b25 163-5°, n20D 1.4573; picrate, m. 123°) results in 79.4% yield from I and H2N(CH2)3NEt2 and in 76.4% yield from Br(CH2)2CN and H2N(CH2)3NEt2. The reaction is reversible. When the higher β-dialkylaminopropionitriles are heated near their b. p. for any length of time, they slowly decompose to give some of the R2NH. (HOCH2CH2)2NH (II) and I, allowed to stand 8 h. at room temperature, give (HOCH2CH2)2NCH2CH2CN (III), which could be isolated as the picrate, m. 108-9°; however, attempted distillation gave 67% of III and a nondistillable polymer. 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EtNH2, warmed for 24 h. at 50° and allowed to stand 2 days at room temperature give 97% of Et2NCH2CH2CN (VII), b735 196°, n 1.4356; picrate, m. 85°; by using mol. ratios and heating on the steam bath for 8 h., the yield is 74%; 5 mols. of EtNH2 and 9.5 mols. of com. I, refluxed 0.5 h. and kept at 0° overnight, give 93% of VII. In general, better yields result if the reaction product is allowed to reach equilibrium at room temperature before distillation; the product should be distilled more or less rapidly at a low temperature if possible. β-Ethylaminopropionitrile, b30 92°, n 1.4318, 90.4%; picrate, m. 163°. Bis(2-cyanoethyl)ethylamine, b30 200-2°, n 1.4591, 60%; picrate, m. 170°. Pr2NCH2CH2CN, b20 116°, n 1.4381, 88%; picrate, m. 111°. Bu2NCH2CH2CN, b20 141°, n 1.4423, 91%; picrate, m. 75°. β-Diamylaminopropionitrile, b19 159-61°, n 1.4457, 89%; the picrate is an oil. β-Dihexylaminopropionitrile, b2 145-6°, n 1.4483, 85%. β-(1-Piperidyl)-2-propionitrile, b30 129-30°, n 1.4697, 93°; picrate, m. 160°. β-(4-Morpholinyl)propionitrile, b20 149°, n 1.4710, 95%; picrate, m. 139.5°. Bis(2-cyanoethyl)ethanolamine picrate, m. 137-8°. 1-Diethylamino-3-[bis(2-cyanoethyl)amino]propane, b25 233-5°, n 1.4709, 8.8%; picrate, m. 166-7°. I and (Et2NCH2CH2CH2)2NH with 0.1 g. Cu bronze, heated at 100° for 24 h. and allowed to stand 24 h. at room temp., give 78% of β-[bis(3-diethylaminopropyl)amino]propionitrile, b3 153°, n 1.4640; picrate, m. 157-8°. β-[2-(4-Morpholinyl)ethylamino]propionitrile, b20 183°, n 1.4817, 81.5%; picrate, m. 176.5°; β-[3-(4-morpholinyl)propylamino]propionitrile, b9 178-80°, n 1.4819, 76%; picrate, m. 148-9°. β-(2-Diethylaminoethoxy)propionitrile, prepd. in 80% yield by adding 100 g. I to 220 g. Et2NCH2CH2OH and 2.3 g. MeONa during 0.5 h. at 25°, allowing to stand overnight, treating with 4.2 mL. concd. H2SO4 and distg. the filtrate, b25 145°, n 1.4430; picrate, m. 75°; β-(3-diethylaminopropoxy)propionitrile, b25 148-50°, n 1.4440, 75.4%. β-(4-Diethylamino-1-methylbutoxy)propionitrile, b3 125-30°, n 1.4456, 66%. I (27 g.) and 53 g. PhNHMe do not react when heated at 180° for 4 h.; in the presence of 1 g. CuSO4.5H2O there results 20 g. of β-(methylphenylamino)propionitrile, b20 175-7°; no reaction occurs with PhCH2NMe3OH in dioxane at 100°; picrate, m. 118°. I (250 mL.) and 167 g. carbazole, cooled in an ice-bath and treated with 2 cc. 40% PhCH2NMe3OH, with heating on the steam bath for 1 h., give 85.4% of 9-(2-cyanoethyl)carbazole, m. 155.5°. I and tetrahydroquinoline did not react with the usual catalysts at 160° after 4 h.; addn. of 100 g. I to 133 g. of tetrahydroquinoline at 125°, with refluxing for 6 h., gives 75.5% of 1-(2-cyanoethyl)tetrahydroquinoline, b10 192°, n 1.5780; picrate, m. 172°. γ-Diethylaminobutyronitrile, b21 101-3°, n 1.4351, 86%; picrate, m. 69-70°; γ-(1-piperidyl)butyronitrile, b25 127-9°, n 1.4653, 87%; picrate, m. 117°; γ-(4-morpholinyl)butyronitrile, b25 148-50°, n 1.4665, 70%; picrate, m. 152-3°. The redn. of the nitriles to amines was carried out with approx. 10 g. Raney Ni per mol. of nitrile at temps. of 90-130° and H pressures of 67 to 270 atm. The formation of secondary amines (5-32%) was obsd., being greater in the case of the γ-aminobutyronitriles; the redn. can be controlled and the yield of primary amine increased by carrying out the hydrogenation in the presence of NH3; the yield of secondary amines can be raised by reducing the nitrile in the presence of an excess of the primary amine. It is believed that the yields of primary products can be increased in almost every case if H pressures of 250-300 atm. are employed and larger amts. of NH3 are introduced. The original gives the pressure, temp., time and solvent used for the various redns. The following compds. are reported. (CH2)3(NH2)2, b735 138°, n 1.4600, 23%; picrate, m. 178°. Et2N(CH2)3NH2, b735 168°, n 1.4355 (picrate, m. 194°), yields up to 72%; bis(3-diethylaminopropyl)amine, b3 107°, n 1.4541 (picrate, m. 153-4°), yields up to 29%. 3-Ethylaminopropylamine, b735 156°, n 1.4441, 74%; picrate, m. 193°. Bis(3-aminopropyl)ethylamine, b20 135°, n 1.4709, 16%; picrate, m. 197-9°. Pr2N(CH2)3NH2, b20 94°, n 1.4435, 49%; picrate, m. 181°; Bu2N(CH2)3NH2, b20 121°, n 1.4462, 32%; picrate, m. 188°. 3-(1-Piperidyl)propylamine, b730 205°, n 1.4750, 68.5%; picrate, m. 209-10°. Bis[3-(1-piperidyl)propyl]amine, b2 153°, n 1.4916, 10%; picrate, m. 193°. 3-(4-Morpholinyl)propylamine, b733 219°, n 1.4762, 70.6%; picrate, m. 166°. Bis[3-(4-morpholinyl)propyl]amine, b5 185°, n 1.4918, 10%; picrate, m. 213-15°; in the prepn. of the primary amine, up to 35% morpholine are formed under certain conditions. 4-Et2N(CH2)4NH2, b18 85-8°, n 1.4462, 51%; picrate, m. 155-6°. 4-(1-Piperidyl)butylamine, b25 118-20°, n 1.4756, 53.8%; picrate, m. 160.5°. Bis[4-(1-piperidyl)butyl]amine, b25 220-5°, n 1.4898, 32%; picrate, m. 202-3°. 4-(4-Morpholinyl)butylamine, b20 122°, n 1.4760, 62%; picrate, m. 148°. Bis[4-(4-morpholinyl)butyl]amine, b3 200-2°, n 1.4900, 23.8%; picrate, m. 136°. Bis(3-aminopropyl) ether, b32 113°, n 1.4618, 29%. 3-(3-Diethylaminopropylamino)propylamine, b25 142-4°, n 1.4630, 51%; picrate, m. 197-8°; bis[3-(3-diethylaminopropylamino)propyl]amine, b25 253-60°, n 1.4710, 31%; picrate, m. 197°. 3-[Bis(3-diethylaminopropyl)amino]propylamine b3 155-65°, n 1.4662, 52%; picrate, m. 162.5°. 3-[2-(4-Morpholinyl)ethylamino]propylamine, b2 120-3°, n 1.4870, 57.5%; picrate, m. 208°. 3-[3-(4-Morpholinyl)propylamino]propylamine, b1.5 137-40°, n 1.4878, 45.2%; picrate, m. 205°. 3-(2-Diethylaminoethoxy)propylamine, b25 118-22°, n 1.4498, 56.7°. Bis[3-(2-diethylaminoethoxy)propyl]amine, b3 175°, n 1.4582, 23.8%. 3-(3-Diethylaminopropoxy)propylamine, b25 130-2°, n 1.4500, 57.4%. Bis[3-(3-diethylaminopropoxy)propyl]amine, b3 182°, n 1.4581, 28.2%. 3-(4-Diethylamino-1-methylbutoxy)propylamine, b2 80-3°, n 1.4492, 50.5%; picrate, m. 88-9°. Bis[3-(4-diethylamino-1-methylbutyoxy)propyl]amine, b3 210-15°, n 1.4580, 23%. 3-(Methylphenylamino)propylamine, b40 171-2°, 63%; HBr salt, m. 120°; picrate, m. 189°. 9-(3-Aminopropyl)carbazole, b3 228°, 70.5%; HCl salt, m. 273°; picrate, m. 206-7°. 1-(3-Aminopropylamino)tetrahydroquinoline, b3 132-5°, n 1.5828, 82%. The following N1-sulfanilamide derivs. were prepd. through the N4-Ac derivs.; in some cases the Ac derivs. were viscous sirups which did not crystallize. 3-Diethylaminopropyl, m. 109-10°, 20%; 3-dipropylaminopropyl, m. 98-8.5°, 57°; 3-dibutylaminopropyl, HCl salt, m. 110-15°, 53.5%; 3-(1-piperidyl)propyl, m. 105.5-6°, 63.5% (Ac deriv., m. 109-11°); 3-(4-morpholinyl)propyl, m. 94.5-5°, 79% (Ac deriv., m. 97-8°); bis(3-diethylaminopropyl), HCl salt, m. 195-7°, 66.5% (Ac deriv., m. 83-5°); bis[3-(1-piperidyl)propyl], m. 74-6°, 71%. It was not possible to use C5H5N or C5H5N-Me2CO as solvents in the prepn. of these derivs.

IT Nitriles
(and their hydrogenation or reduction to amines)
IT Reduction
(of nitriles to amines)
IT Amines
(preparation of)
IT 1(2)-Quinolinepropionitrile, 3,4-dihydro-
1(2)-Quinolinepropionitrile, 3,4-dihydro-, picrate
1,3-Propanediamine, picrate
1,3-Propanediamine, N,N-dipropyl-, dipicrate
1,3-Propanediamine, N-(3-aminopropyl)-N-(3-diethylaminopropyl)-N',N'-diethyl-, picrate
1,3-Propanediamine, N-(3-aminopropyl)-N-ethyl-, picrate
1,3-Propanediamine, N-(3-diethylaminopropyl)-N'-[3-(3-diethylaminopropylamino)propyl]-
1,3-Propanediamine, N-(3-diethylaminopropyl)-N'-[3-(3-diethylaminopropylamino)propyl]-, picrate
1,3-Propanediamine, N-ethyl-, picrate
1,3-Propanediamine, N'-(3-aminopropyl)-N,N-diethyl-, picrate
1-Piperidinebutyronitrile, picrate
1-Piperidinepropionitrile, picrate
3,7,11,15,19-Pentazaheneicosane, 3,19-diethyl-
3,7,11,15,19-Pentazaheneicosane, 3,19-diethyl-, picrate
3,7-Diazadecan-10-amine, 3-ethyl-
4-Morpholinepropionitrile, picrate
6,14-Dioxa-3,10,17-triazanonadecane, 3,17-diethyl-
7,15-Dioxa-3,11,19-triazaheneicosane, 3,19-diethyl-
8,16-Dioxa-3,12,21-triazatricosane, 3,21-diethyl-7,17-dimethyl-
9-Carbazolepropionitrile
Amylamine, 4,4'-[iminobis(trimethyleneoxy)]bis[N,N-diethyl-
Amylamine, 4-(3-aminopropoxy)-N,N-diethyl-
Amylamine, 4-(3-aminopropoxy)-N,N-diethyl-, picrate
Butyronitrile, γ-diethylamino-, picrate
Dibutylamine, 4,4'-di-1-piperidyl-
Dibutylamine, 4,4'-di-1-piperidyl-, picrate
Dibutylamine, 4,4'-di-4-morpholinyl-
Dibutylamine, 4,4'-di-4-morpholinyl-, picrate
Diethylamine, N-[3-(3-aminopropoxy)propyl]-
Dipropylamine, 3,3'-bis(3-diethylaminopropoxy)-
Dipropylamine, 3,3'-bis(3-diethylaminopropylamino)-
Dipropylamine, 3,3'-bis(3-diethylaminopropylamino)-, picrate
Dipropylamine, 3,3'-bis[4-diethylamino-1-methylbutoxy)-
Dipropylamine, 3,3'-bis(diethylamino)-, picrate
Dipropylamine, 3,3'-di-1-piperidyl-
Dipropylamine, 3,3'-di-1-piperidyl-, picrate
Dipropylamine, 3,3'-di-4-morpholinyl-
Dipropylamine, 3,3'-di-4-morpholinyl-, picrate
Dipropylamine, 3,3'-diamino-N-ethyl-, picrate
Ethanol, 2,2'-(3-aminopropylimino)di-, picrate
Morpholine, 4,4'-(iminoditetramethylene)di-
Morpholine, 4,4'-(iminoditetramethylene)di-, picrate
Morpholine, 4,4'-(iminoditrimethylene)di-
Morpholine, 4,4'-(iminoditrimethylene)di-, picrate
Morpholine, 4-(4-aminobutyl)-
Morpholine, 4-(4-aminobutyl)-, picrate
Morpholine, 4-[2-(3-aminopropylamino)ethyl]-
Morpholine, 4-[2-(3-aminopropylamino)ethyl]-, picrate
Morpholine, 4-[3-(3-aminopropylamino)propyl]-
Morpholine, 4-[3-(3-aminopropylamino)propyl]-, picrate
Piperidine, 1,1'-(iminobistetramethylene)di-
Piperidine, 1,1'-(iminobistetramethylene)di-, picrate
Piperidine, 1,1'-(iminobistrimethylene)di-
Piperidine, 1,1'-(iminobistrimethylene)di-, picrate
Piperidine, 1-(4-aminobutyl)-, picrate
Propionitrile, β,β'-(2-diethylaminoethylimino)di-
Propionitrile, β,β'-(2-diethylaminoethylimino)di-, picrate
Propionitrile, β,β'-(2-hydroxyethylimino)di-
Propionitrile, β,β'-(2-hydroxyethylimino)di-, picrate
Propionitrile, β,β'-(ethylimino)di-
Propionitrile, β,β'-(ethylimino)di-, picrate
Propionitrile, β-(2-diethylaminoethoxy)-
Propionitrile, β-(2-diethylaminoethoxy)-, picrate
Propionitrile, β-(3-diethylaminopropoxy)-
Propionitrile, β-(3-diethylaminopropylamino)-
Propionitrile, β-(3-diethylaminopropylamino)-, picrate
Propionitrile, β-(4-diethylamino-1-methylbutoxy)-

Propionitrile, β-[2-(4-morpholinyl)ethylamino]-
Propionitrile, β-[2-(4-morpholinyl)ethylamino]-, picrate
Propionitrile, β-[3-(4-morpholinyl)propylamino]-
Propionitrile, β-[3-(4-morpholinyl)propylamino]-, picrate
Propionitrile, β-[bis(2-hydroxyethyl)amino]-
Propionitrile, β-[bis(2-hydroxyethyl)amino]-, picrate
Propionitrile, β-[bis(3-diethylaminopropyl)-amino]-
Propionitrile, β-[bis(3-diethylaminopropyl)-amino]-, picrate
Propionitrile, β-amino-, picrate
Propionitrile, β-diamylamino-
Propionitrile, β-diamylamino-, picrate
Propionitrile, β-dibutylamino-
Propionitrile, β-dibutylamino-, picrate
Propionitrile, β-diethylamino-
Propionitrile, β-diethylamino-, picrate
Propionitrile, β-dihexylamino-
Propionitrile, β-dipropylamino-
Propionitrile, β-dipropylamino-, picrate
Propionitrile, β-ethylamino-
Propionitrile, β-ethylamino-, picrate
Propionitrile, β-N-methylanilino-
Propionitrile, β-N-methylanilino-, picrate
Propylamine, 3-(3-diethylaminopropoxy)-
Putrescine, N,N-dibutyl-
Putrescine, N,N-dibutyl-, picrate
Putrescine, N,N-diethyl-, picrate
Sulfanilamide, N1,N1-bis[3-(1-piperidyl)propyl]-
Sulfanilamide, N1-(3-dibutylaminopropyl)-, hydrochloride
Sulfanilamide, N1-(3-diethylaminopropyl)-
Sulfanilamide, N1-(dipropylaminopropyl)-
Sulfanilamide, N1-[3-(1-piperidyl)propyl]-
Sulfanilamide, N1-[3-(1-piperidyl)propyl]-, acetyl deriv.
Sulfanilamide, N1-[3-(4-morpholinyl)propyl]-
Sulfanilamide, N1-[3-(4-morpholinyl)propyl]-, acetyl deriv.
Triethylamine, 2,2'''-(iminoditrimethylenedioxy)bis-
Triethylamine, 2-(3-aminopropoxy)-
Tripropylamine, 3-amino-3',3''-bis(diethylamino)-
Tripropylamine, 3-amino-3',3''-bis(diethylamino)-, picrate

IT Aniline, N-(3-aminopropyl)-N-methyl-
(and derivs.)

IT Butyronitrile, γ-amino-
Sulfanilamide, N1,N1-bis(3-diethylaminopropyl)-
(derivs.)

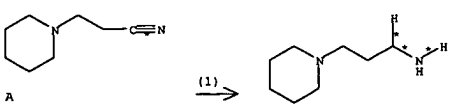
IT 23690-10-0, Carbazole, 9-(3-aminopropyl)- 53485-07-7,
1,3-Propanediamine, N-methyl-N-phenyl-
(and derivs.)

IT 107-12-0, Propionitrile
(derivs.)

IT 104-78-9, 1,3-Propanediamine, N,N-diethyl- 109-76-2, 1,3-Propanediamine
111-94-4, Propionitrile, β,β'-iminodi- 123-00-2, Morpholine,
4-(3-aminopropyl)- 151-18-8, Propionitrile, β-amino- 2157-24-6,
Propylamine, 3,3'-oxybis- 2372-77-2, Dipropylamine,
3,3'-diamino-N-ethyl-
2637-31-2, Quinoline, 1-(3-aminopropyl)-1,2,3,4-tetrahydro-
3088-41-3, 1-Piperidinepropionitrile 3529-08-6, Piperidine,
1-(3-aminopropyl)- 4542-47-6, 4-Morpholinepropionitrile 4672-18-8,
1-Piperidinebutyronitrile 4985-85-7, Ethanol, 2,2'-(3-
aminopropylimino)di- 5336-75-4, Butyronitrile, γ-diethylamino-
6050-28-8, Dipropylamine, 3,3'-bis(diethylamino)- 6345-82-0,

1,3-Propanediamine, N,N-dipropyl- 7505-16-0, 1,3-Propanediamine,
N,N-diethyl-, picrate 7528-78-1, Propionitrile, β,β',β''-
nitrilotri- 10563-23-2, 1,3-Propanediamine, N-ethyl- 27431-62-5,
Putrescine, N,N-diethyl- 34155-25-4, Propylamine, 3-(2-
diethylaminoethoxy)- 74247-30-6, Piperidine, 1-(4-aminobutyl)-
103502-67-6, 1,3-Propanediamine, N'-(3-aminopropyl)-N,N-diethyl-
105788-80-5, Morpholine, 4-(3-aminopropyl)-, picrate 106838-31-7,
Piperidine, 1-(3-aminopropyl)-, picrate 334972-85-9,
1,3-Propanediamine,
N-(3-aminopropyl)-N-(3-diethylaminopropyl)-N',N'-diethyl- 760205-17-2,
Dipropylamine, 3,3'-bis(2-diethylaminoethoxy)-
(prepn. of)

RX(1) OF 3 A ===> B

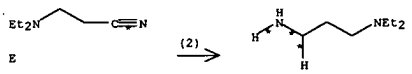


A B

YIELD 45%

RX(1) RCT A 3088-41-3
RGT C 1333-74-0 H2
PRO B 3529-08-6
CAT 7440-02-0 Ni
NTE Classification: Hydrogenation; Reduction; # Conditions: /H2
Raney Nickel; 35mn 120 deg /109atm

RX(2) OF 3 E ===> F

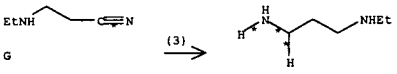


E F

YIELD 70%

RX(2) RCT E 5351-04-2
RGT C 1333-74-0 H2
PRO F 104-78-9
CAT 7440-02-0 Ni
NTE Classification: Hydrogenation; Reduction; # Conditions: /H2 Ni
NH3; 130 deg /85atm

RX(3) OF 3 G ===> H



G H

YIELD 74%

RX(3) RCT G 21539-47-9
RGT C 1333-74-0 H2
PRO H 10563-23-2
CAT 7440-02-0 Ni
SOL 64-17-5 EtOH
NTE Classification: Hydrogenation; Reduction; # Conditions: /H2
Raney Nickel; NH3 EtOH; /100atm 10mn 100 deg; # Comments:
Numerous examples

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NOTICE SET TO OFF  FOR DISPLAY COMMAND
SET COMMAND COMPLETED

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                                                         ENTRY    SESSION
FULL ESTIMATED COST                                       7.05     175.25

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                                                         ENTRY    SESSION
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L1              STRUCTURE UPLOADED
L2            6 S L1
L3           70 S L1 FULL

     FILE 'CAPLUS' ENTERED AT 17:35:25 ON 21 JAN 2005
L4           70 S L3
L5       787048 S NI OR NICKEL
L6       938306 S CO OR COBALT
L7           16 S L4 AND L5
L8            9 S L4 AND L6
L9           20 S L7 OR L8

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               SET NOTICE LOGIN DISPLAY

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COST IN U.S. DOLLARS                                    SINCE FILE      TOTAL
                                                           ENTRY    SESSION
FULL ESTIMATED COST                                         4.50     179.75

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)              SINCE FILE      TOTAL
                                                           ENTRY    SESSION
CA SUBSCRIBER PRICE                                         0.00     -15.28

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FILE CONTENT:1840 - 16 Jan 2005 VOL 141 ISS 20

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     FILE 'CASREACT' ENTERED AT 17:34:38 ON 21 JAN 2005
L1              STRUCTURE UPLOADED
L2            6 S L1
L3           70 S L1 FULL

     FILE 'CAPLUS' ENTERED AT 17:35:25 ON 21 JAN 2005
L4           70 S L3
L5       787048 S NI OR NICKEL
L6       938306 S CO OR COBALT
L7           16 S L4 AND L5
L8            9 S L4 AND L6
L9           20 S L7 OR L8

     FILE 'CASREACT' ENTERED AT 17:36:53 ON 21 JAN 2005
               SET NOTICE DISPLAY 1
               SET NOTICE LOGIN DISPLAY

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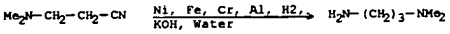
L3 ANSWER 1 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

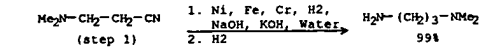
RX(1) OF 1



REF: U.S. Pat. Appl. Publ., 2004147784, 29 Jul 2004
NOTE: Sponge nickel was used, described app., optimization study, reaction run in 6 cycles each cycle was 27 min

L3 ANSWER 2 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

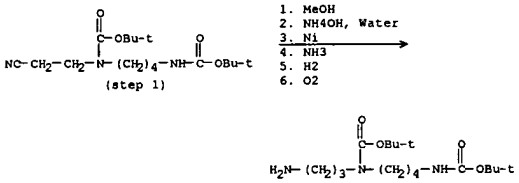
RX(1) OF 1



REF: PCT Int. Appl., 2004060853, 22 Jul 2004
NOTE: Sponge nickel was used, autoclave was used

L3 ANSWER 3 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

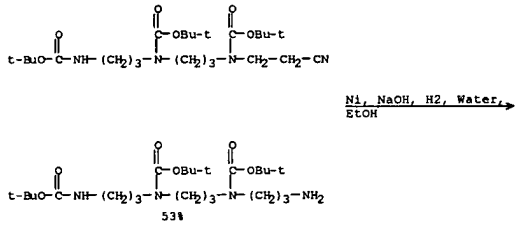
RX(8) OF 385



REF: Journal of Organic Chemistry, 69(10), 3530-3537; 2004

L3 ANSWER 4 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

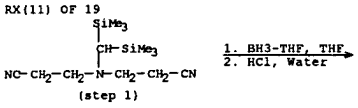
RX(37) OF 116



REF: Journal of Medicinal Chemistry, 46(26), 5712-5724; 2003
NOTE: Raney nickel used

L3 ANSWER 5 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(11) OF 19



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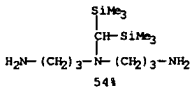
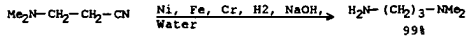


REF: Journal of Organometallic Chemistry, 686(1-2), 306-312; 2003

L3 ANSWER 6 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

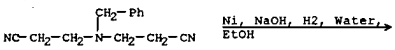
RX(1) OF 1

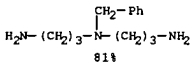


REF: U.S., 6660887, 09 Dec 2003

L3 ANSWER 7 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(16) OF 149



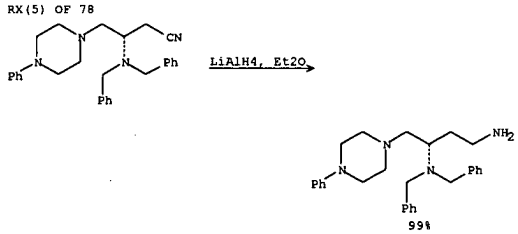


REF: Journal of the American Chemical Society, 125(40), 12196-12210; 2003

NOTE: Raney nickel used, safety, explosion hazard, air must be completedly replaced by hydrogen in the Parr app. before reaction is begun

L3 ANSWER 8 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

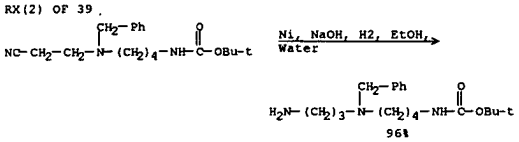
RX(5) OF 78



REF: Bioorganic & Medicinal Chemistry Letters, 13(5), 851-854; 2003

L3 ANSWER 9 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

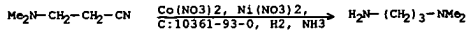
RX(2) OF 39



REF: Tetrahedron, 59(11), 2007-2014; 2003

L3 ANSWER 10 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

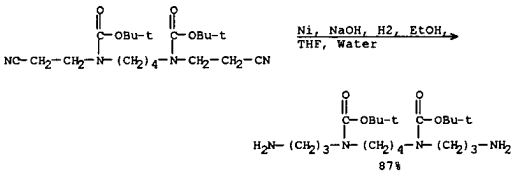
RX(2) OF 2



REF: Ger. Offen., 10152135, 30 Apr 2003
NOTE: catalyst prepd. prior to use, catalyst adsorbed on aluminosilicate, high pressure

L3 ANSWER 11 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

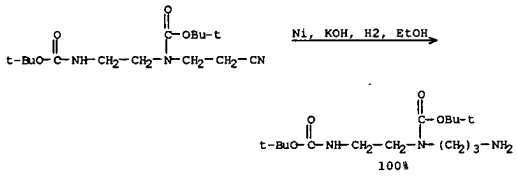
RX(9) OF 24



REF: Bioorganic & Medicinal Chemistry, 11(1), 87-94; 2003

L3 ANSWER 12 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

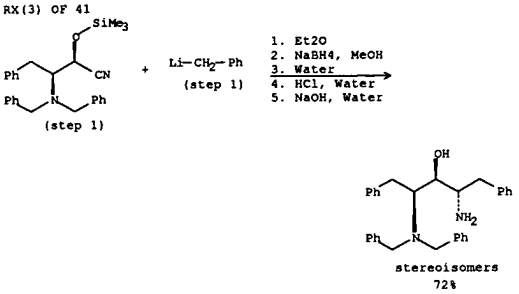
RX(16) OF 215



REF: Bioorganic & Medicinal Chemistry, 11(2), 235-249; 2003
NOTE: Raney nickel used

L3 ANSWER 13 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

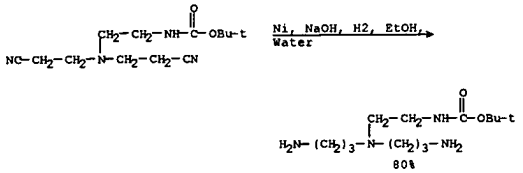
RX(3) OF 41



REF: Journal of Organic Chemistry, 68(4), 1418-1425; 2003
NOTE: stereoselective, KEY STEP, anaerobic 1st and 2nd stages

L3 ANSWER 14 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

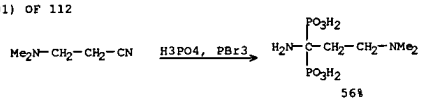
RX(3) OF 124



REF: European Journal of Medicinal Chemistry, 37(7), 541-551; 2002

L3 ANSWER 15 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

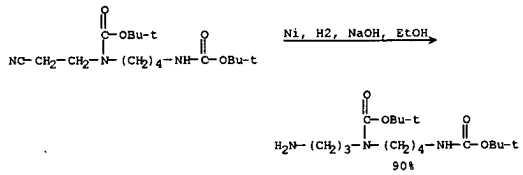
RX(101) OF 112



REF: Journal of Medicinal Chemistry, 45(17), 3721-3738; 2002

L3 ANSWER 16 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

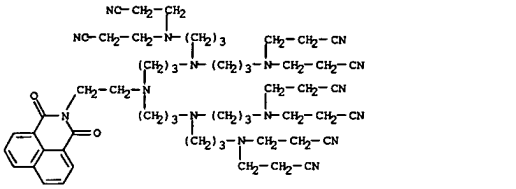
RX(12) OF 114



REF: Journal of the Chemical Society, Perkin Transactions 1, (8), 1115-1123; 2002
NOTE: Raney nickel used

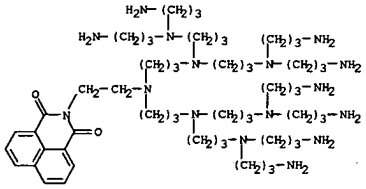
L3 ANSWER 17 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(7) OF 31



Ni, H2, NaOH, EtOH

RX(7) OF 31

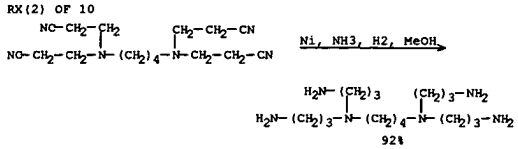


52%

REF: Bioorganic & Medicinal Chemistry Letters, 11(23), 3027-3029; 2001
NOTE: Raney nickel used

L3 ANSWER 18 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(2) OF 10



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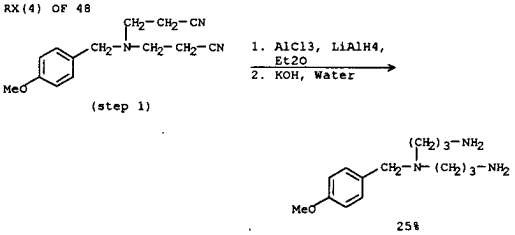
REF: Jingxi Huagong, 18(7), 414-416, 420; 2001

L3 ANSWER 19 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(4) OF 48



(step 1)

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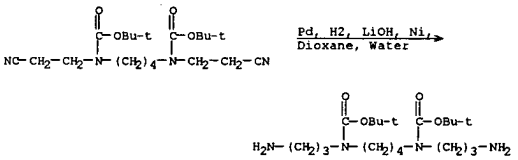
REF: Molecules [online computer file], 6(3), 230-243; 2001

L3 ANSWER 20 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(15) OF 138

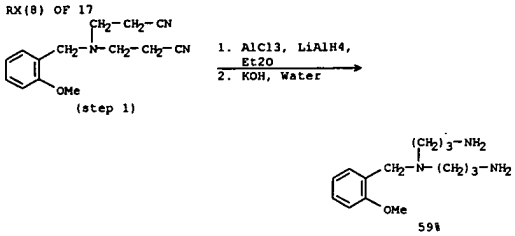


94%

REF: Journal of Medicinal Chemistry, 44(21), 3440-3452; 2001
NOTE: catalyst on carbon, Raney nickel used

L3 ANSWER 21 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

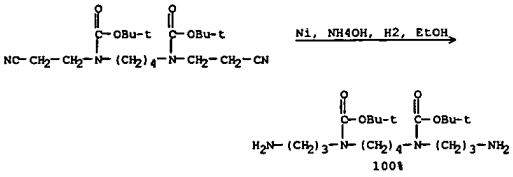
RX(8) OF 17



REF: European Journal of Organic Chemistry, (16), 3119-3125; 2001
NOTE: no solvent

L3 ANSWER 22 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

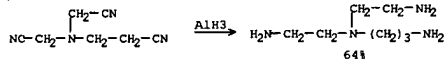
RX(16) OF 75



REF: Farmaco, 56(1-2), 127-131; 2001
NOTE: Raney Nickel used

L3 ANSWER 23 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

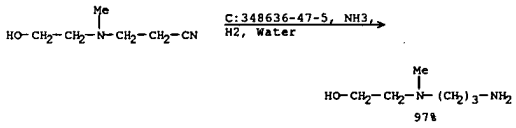
RX(1) OF 20



REF: European Journal of Inorganic Chemistry, (5), 1279-1285; 2001
NOTE: literature prepn.

L3 ANSWER 24 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

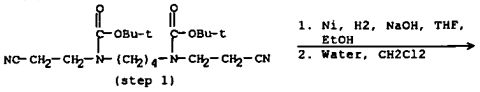
RX(1) OF 1



REF: Jpn. Kokai Tokkyo Koho, 2001187766, 10 Jul 2001

L3 ANSWER 25 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(1) OF 10



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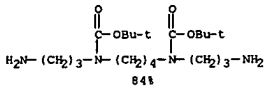
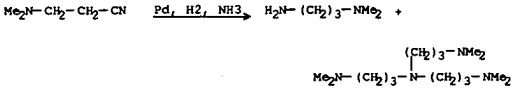


84%

REF: Journal of Organic Chemistry, 66(7), 2480-2483; 2001

L3 ANSWER 26 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(1) OF 1

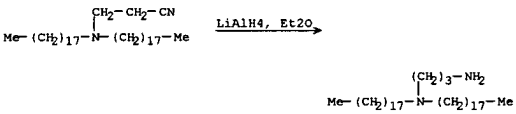


REF: Collection of Czechoslovak Chemical Communications, 65(11), 1805-1819; 2000

NOTE: by products produced

L3 ANSWER 27 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

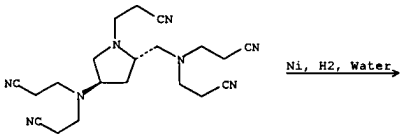
RX(2) OF 17



REF: Bioconjugate Chemistry, 12(1), 56-61; 2001

L3 ANSWER 28 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(4) OF 60



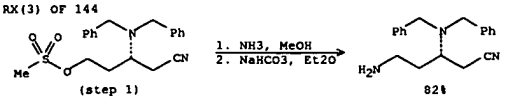
92%

REF: Organic Letters, 3(1), 103-106; 2001

NOTE: Raney nickel used

L3 ANSWER 29 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(3) OF 144

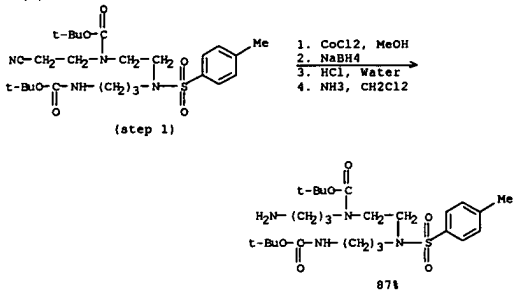


(step 1) 82%

REF: Journal of Organic Chemistry, 65(22), 7406-7416; 2000
NOTE: stereoselective

L3 ANSWER 30 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(6) OF 95

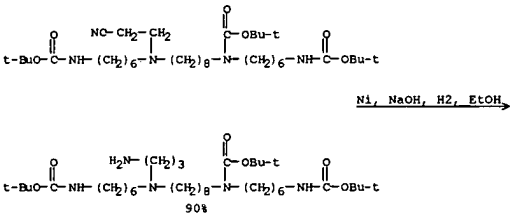


(step 1) 87%

REF: Tetrahedron, 56(27), 4759-4764; 2000

L3 ANSWER 31 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(39) OF 136



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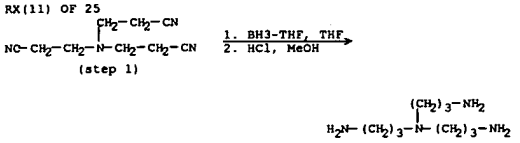
REF: Journal of Medicinal Chemistry, 42(25), 5212-5223; 1999

L3 ANSWER 32 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

L3 ANSWER 33 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(11) OF 25

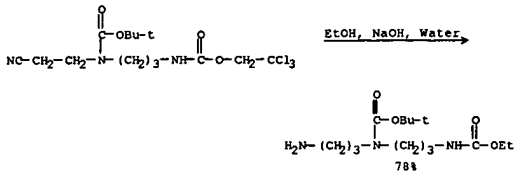


(step 1)

REF: Journal of the American Chemical Society, 119(24), 5638-5647; 1997
NOTE: CLAIMED TO BE SIGNIFICANT IMPROVEMENT OVER RANEY-NI PROCESSES

L3 ANSWER 34 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

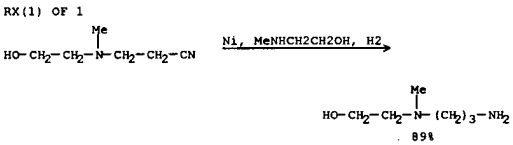
RX(11) OF 41



REF: Journal of Chemical Research, Synopses, (8), 366-367; 1996

L3 ANSWER 35 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

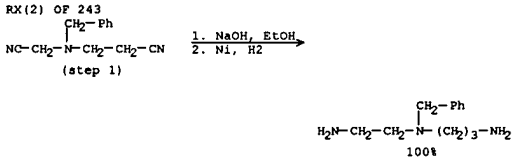
RX(1) OF 1



REF: Jpn. Kokai Tokkyo Koho, 07157453, 20 Jun 1995, Heisei

L3 ANSWER 36 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(2) OF 243

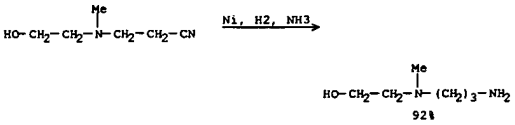


(step 1)

REF: Tetrahedron, 51(4), 1197-208; 1995
NOTE: RANEY NICKEL IN SECOND STAGE

L3 ANSWER 37 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

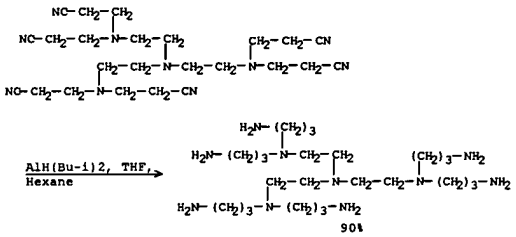
RX(1) OF 1



REF: Jpn. Kokai Tokkyo Koho, 05246959, 24 Sep 1993, Heisei

L3 ANSWER 38 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

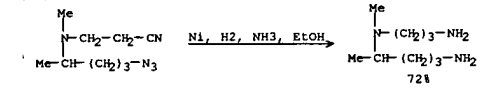
RX(2) OF 4



REF: Chemische Berichte, 126(9), 2133-5; 1993

L3 ANSWER 39 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

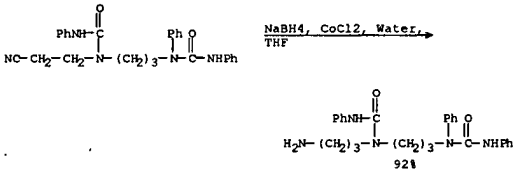
RX(22) OF 58



REF: Journal of Organic Chemistry, 58(14), 3736-41; 1993
NOTE: Raney nickel

L3 ANSWER 40 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

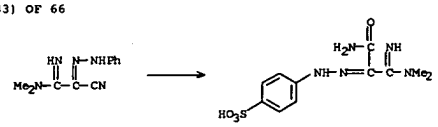
RX(2) OF 11



REF: Journal of Organic Chemistry, 57(14), 3763-5; 1992

L3 ANSWER 41 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(33) OF 66



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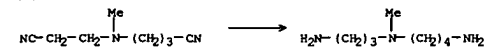
REF: Monatshefte fuer Chemie, 122(3), 195-207; 1991

L3 ANSWER 42 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

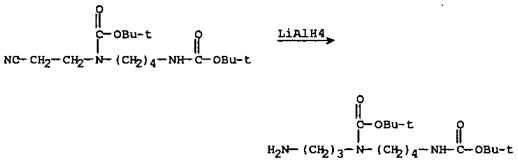
RX(2) OF 54



REF: Eur. Pat. Appl., 376819, 04 Jul 1990

L3 ANSWER 43 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(5) OF 84



REF: Pure and Applied Chemistry, 62(7), 1223-30; 1990

L3 ANSWER 44 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

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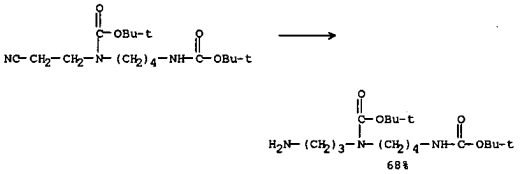

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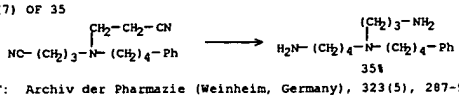
REF: Tetrahedron, 46(9), 3267-86; 1990

L3 ANSWER 45 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(7) OF 35



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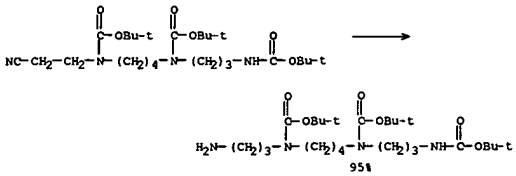
REF: Archiv der Pharmazie (Weinheim, Germany), 323(5), 287-94; 1990

L3 ANSWER 46 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(8) OF 54



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REF: Journal of the American Chemical Society, 112(18), 6696-704; 1990

L3 ANSWER 47 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

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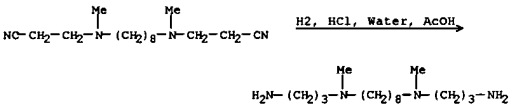


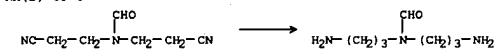
4 HCl

7%

REF: Journal of Medicinal Chemistry, 33(5), 1369-75; 1990

L3 ANSWER 48 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

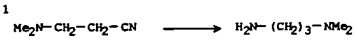
RX(2) OF 5



REF: Ger. Offen., 3811342, 12 Oct 1989

L3 ANSWER 49 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

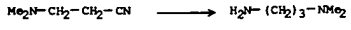
RX(1) OF 1



REF: Eur. Pat. Appl., 316761, 24 May 1989

L3 ANSWER 50 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

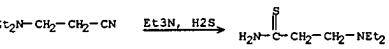
RX(1) OF 1



REF: Khimicheskaya Tekhnologiya (Kiev), (4), 58-61; 1989

L3 ANSWER 51 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(1) OF 1



REF: U.S.S.R., 1438831, 23 Nov 1988

L3 ANSWER 52 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

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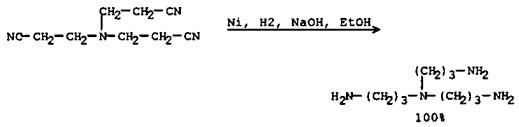
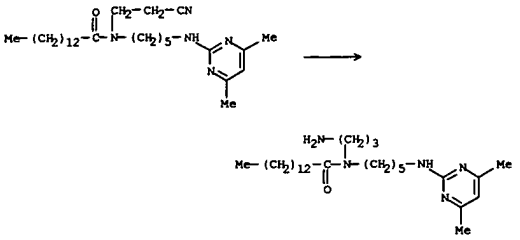


REF: Journal of the American Chemical Society, 111(1), 186-90; 1989

L3 ANSWER 53 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(4) OF 15



REF: U.S., 4762949, 09 Aug 1988

L3 ANSWER 54 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

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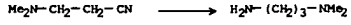
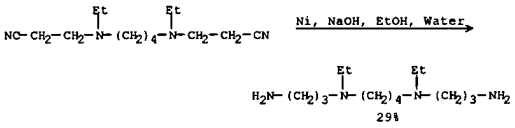


REF: Chemiker-Zeitung, 111(4), 117-25; 1987

L3 ANSWER 55 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

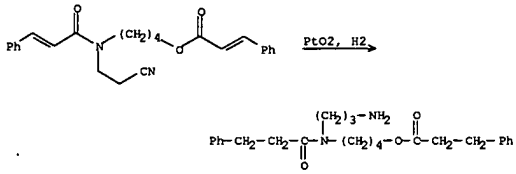
RX(11) OF 36



REF: Journal of Medicinal Chemistry, 31(6), 1183-90; 1988

L3 ANSWER 56 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

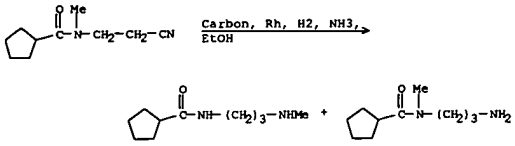
RX(3) OF 138



REF: Tetrahedron Letters, 27(2), 207-10; 1986

L3 ANSWER 57 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(29) OF 127



REF: Journal of Medicinal Chemistry, 29(1), 19-25; 1986

L3 ANSWER 58 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

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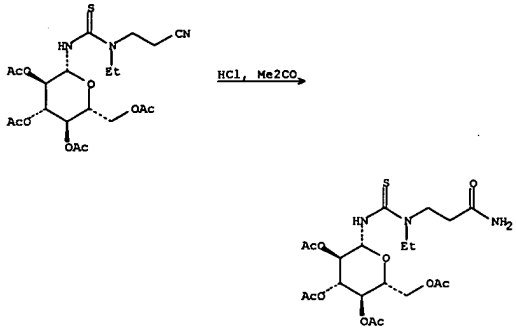
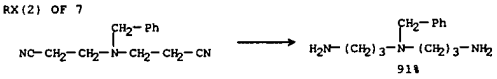


REF: Synthesis, (6-7), 686-8; 1985

L3 ANSWER 59 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(2) OF 7



REF: Synthesis, (9), 782-4; 1984

L3 ANSWER 60 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

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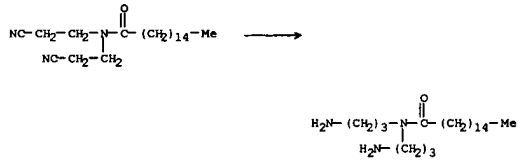

REF: Journal of Pharmaceutical Sciences, 70(8), 956-9; 1981

L3 ANSWER 61 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

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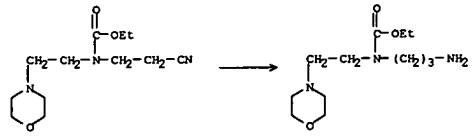
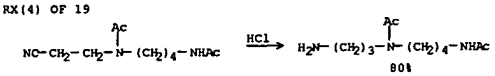


REF: Indian Journal of Chemistry, Section B: Organic Chemistry Including Medicinal Chemistry, 20B(2), 170-1; 1981

L3 ANSWER 62 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

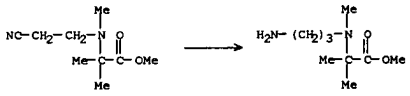
RX(4) OF 19



REF: Polish Journal of Chemistry, 52(11), 2251-4; 1978

L3 ANSWER 63 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

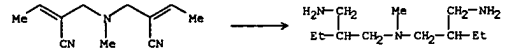
RX(15) OF 59



REF: Polish Journal of Chemistry, 52(5), 1023-8; 1978

L3 ANSWER 64 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

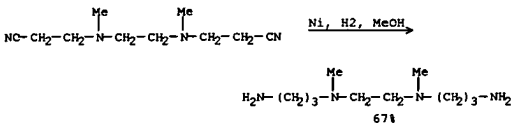
RX(22) OF 47



REF: Journal of Organic Chemistry, 43(4), 622-6; 1978

L3 ANSWER 65 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

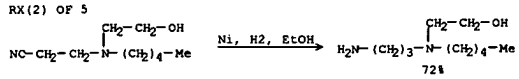
RX(2) OF 10



REF: Monatshefte fuer Chemie, 95(3), 922-41; 1964
NOTE: Classification: Hydrogenation; Reduction; # Conditions: H2 Raney Ni; MeOH; 80-90atm 90-100 deg; 15h

L3 ANSWER 66 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

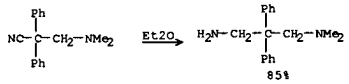
RX(2) OF 5



REF: Journal of the American Chemical Society, 80,, 451-5; 1958
NOTE: Classification: Hydrogenation; Reduction; # Conditions: /H2 Raney Nickel; EtOH NH3; 100 deg /70atm; 1h30mn

L3 ANSWER 67 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

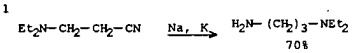
RX(1) OF 4



REF: Journal of the American Chemical Society, 75,, 292-4; 1953
NOTE: Classification: Reduction; # Conditions: LiAlH4 Et2O; /N2 ice bath 3h

L3 ANSWER 68 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

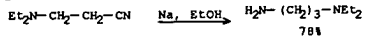
RX(1) OF 1



REF: Zhurnal Obshchei Khimii, 17,, 105-8; 1947
NOTE: Classification: Reduction; # Conditions: Na K n-BuOH; # Comments: Also CA, 42, 112a (1948); Yield range 65-70%

L3 ANSWER 69 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

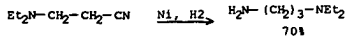
RX(1) OF 1



REF: J. Indian Chem. Soc., 23,, 224-8; 1946
NOTE: Classification: Reduction; # Conditions: Na EtOH; # Comments: Also CA, 41, 2420a (1947)

L3 ANSWER 70 OF 70 CASREACT COPYRIGHT 2005 ACS on STN

RX(2) OF 3



REF: Journal of the American Chemical Society, 66,, 725-31; 1944
NOTE: Classification: Hydrogenation; Reduction; # Conditions: /H2 Ni NH3; 130 deg /85atm

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COST IN U.S. DOLLARS                                    SINCE FILE      TOTAL
                                                           ENTRY    SESSION
FULL ESTIMATED COST                                        190.34     370.09

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FILE LAST UPDATED: 20 Jan 2005  (20050120/ED)

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L3           70 S L1 FULL

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L4           70 S L3
L5       787048 S NI OR NICKEL
L6       938306 S CO OR COBALT
L7           16 S L4 AND L5
L8            9 S L4 AND L6
L9           20 S L7 OR L8

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=> d l4 1-70 abs ibib

L4 ANSWER 1 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB A low-pressure hydrogenation process for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile in the presence of a catalyst system comprising sponge nickel and aqueous alkali is described.

ACCESSION NUMBER: 2004:609968 CAPLUS
DOCUMENT NUMBER: 141:140075
TITLE: Low-pressure hydrogenation process for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile in the presence of a catalyst system comprising sponge nickel and aqueous alkali
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): USA
SOURCE: U.S. Pat. Appl. Publ., 8 pp., Cont.-in-part of U.S. Ser. No. 327,765.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004147784	A1	20040729	US 2003-731733	20031209
US 6660887	B1	20031209	US 2002-327765	20021223
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-327765 A2 20021223
US 2003-731733 A 20031209

OTHER SOURCE(S): CASREACT 141:140075

L4 ANSWER 2 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB A low-pressure hydrogenation process for the production of 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile (I) comprises: feeding hydrogen and 3-(dimethylamino)propionitrile into a low-pressure reactor containing a sponge nickel catalyst, at least one Group IA alkali metal hydroxide (e.g., potassium hydroxide), and water to form a reaction medium; heating the reaction medium to 70-100°; pressurizing the reactor to 45-500 psig; and hydrogenating the nitrile to form I.

ACCESSION NUMBER: 2004:589527 CAPLUS
DOCUMENT NUMBER: 141:123405
TITLE: Low-pressure catalytic hydrogenation process for the manufacture of 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): Solutia Inc., USA
SOURCE: PCT Int. Appl., 20 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

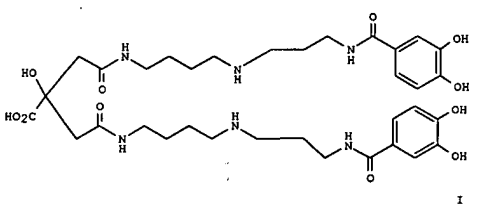
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 6660887	B1	20031209	US 2002-327765	20021223
US 2004147784	A1	20040729	US 2003-731733	20031209

PRIORITY APPLN. INFO.: US 2002-327765 A 20021223
US 2003-731733 A 20031209

OTHER SOURCE(S): CASREACT 141:123405

L4 ANSWER 3 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
GI



I

AB A modular synthesis was developed to access petrobactin (I), a catechol-containing siderophore isolated from Marinobacter hydrocarbonoclasticus. A range of petrobactin homologs with differing dihydroxybenzamide motifs and in one case an increased number of carbons in the polyamine backbone were also synthesized. As such, these systems represent new isomeric probes to study iron transport properties in M. hydrocarbonoclasticus. The synthesis of I and its homologs and the first biol. study of how these agents influence the growth of Mycobacter hydrocarbonoclasticus are reported. New synthetic methods were developed to overcome issues (imide formation) encountered in earlier syntheses. Both the 1H and 13C NMR of I were consistent with the recently revised structure showing that native petrobactin in fact contains a 3,4-dihydroxybenzene motif rather than a 2,3-dihydroxybenzene motif. The preliminary biol. studies suggested that using the native petrobactin for M. hydrocarbonoclasticus-specific growth stimulation may be a poor strategy for oil-spill cleanup.

ACCESSION NUMBER: 2004:325022 CAPLUS
DOCUMENT NUMBER: 141:54103
TITLE: Total Synthesis of Petrobactin and Its Homologues as Potential Growth Stimuli for Marinobacter hydrocarbonoclasticus, an Oil-Degrading Bacteria
AUTHOR(S): Gardner, Richard Andrew; Kinkade, Rebecca; Wang, Chaojie; Phanstiel, Otto, IV
CORPORATE SOURCE: Department of Chemistry, University of Central Florida, Orlando, FL, 32816-2366, USA
SOURCE: Journal of Organic Chemistry (2004), 69(10), 3530-3537
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 141:54103
REFERENCE COUNT: 36 THERE ARE 36 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 4 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB A process for the production of 3-(dimethylamino)propylamine (I) in high (>99%) purity from 3-(dimethylamino)propionitrile utilizing a low-pressure hydrogenation process is described which comprises contacting the nitrile with hydrogen at low pressure in the presence of a sponge nickel catalyst and ≥1 Goup IA metal hydroxide at 70-100°/45-150 psig. The improvement in the process resides in a combination of carrying out the hydrogenation process at low pressures and temps. in the presence of a catalytic amount of caustic base in order to give a I selectivity of >99.60%.

ACCESSION NUMBER: 2003:961180 CAPLUS
DOCUMENT NUMBER: 140:17730
TITLE: Low-pressure hydrogenation process and catalyst system for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): Solutia Inc., USA
SOURCE: U.S., 7 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6660887	B1	20031209	US 2002-327765	20021223
WO 2004060039	A2	20040722	WO 2003-US29721	20030919
WO 2004060039	A3	20040826		

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 2004147784	A1	20040729	US 2003-731733	20031209
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-327765 A 20021223
US 2003-731733 A 20031209

OTHER SOURCE(S): CASREACT 140:17730

L4 ANSWER 4 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
REFERENCE COUNT: 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 5 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB The indenoisoquinolines are a class of noncamptothecin topoisomerase I inhibitors that display significant cytotoxicity in human cancer cell cultures. They offer a number of potential advantages over the camptothecins, including greater chemical stability, formation of more persistent cleavage complexes, and induction of a unique pattern of DNA cleavage sites. Mol. modeling has suggested that substituents on the indenoisoquinoline lactam nitrogen would protrude out of the DNA duplex in the ternary cleavage complex through the major groove. This indicates that relatively large substituents in that location would be tolerated without compromising biol. activity. As a strategy for increasing the potencies and potential therapeutic usefulness of the indenoisoquinolines, a series of compds. was synthesized containing polyamine side chains on the lactam nitrogen. The rationale for the synthesis of these compds. was that the pos. charged ammonium cations would increase DNA affinity through electrostatic binding to the neg. charged DNA backbone, and the polyamines might also facilitate cellular uptake by utilization of polyamine transporters. The key step in the synthesis involved the condensation of Schiff bases, containing protected amine side chains, with substituted homophthalic anhydrides, to afford cis-3-aryl-4-carboxy-1-isoquinolones. These isoquinolones were then converted to indenoisoquinolines with thionyl chloride. Although monoamines were much more potent than the lead compound, no significant increase in potency was observed through incorporation of addnl. amino groups in the side chain. However, one of the monoamine analogs, which features a bis(2-hydroxyethyl)amino group in the side chain, proved to be one of the most cytotoxic indenoisoquinoline synthesized to date, with a GI50 mean-graph midpoint (MGM) of 0.07 μM in the NIH human cancer cell culture screen, and topoisomerase I inhibitory activity comparable to that of camptothecin. The activity of the compds. thus prepared was compared to (4S)-4-ethyl-4-hydroxy-1H-pyrano[3',4':6,7]indolizino[1,2-b]quinoline-3,14(4H,12H)-dione [(20S)-camptothecin], 2,3-dimethoxy-6-methyl-5H-[1,3]dioxolo[5,6]indeno[1,2-c]isoquinoline-5,12(6H)-dione, 6-(3-aminopropyl)-2,3-dimethoxy-5H-[1,3]dioxolo[5,6]indeno[1,2-c]isoquinoline-5,12(6H)-dione monohydrochloride.
ACCESSION NUMBER: 2003:909303 CAPLUS
DOCUMENT NUMBER: **140:111315**
TITLE: Design, Synthesis, and Biological Evaluation of Indenoisoquinoline Topoisomerase I Inhibitors Featuring Polyamine Side Chains on the Lactam Nitrogen
AUTHOR(S): Nagarajan, Muthukaman; Xiao, Xiangshu; Antony, Smitha; Kohlhagen, Glenda; Pommier, Yves; Cushman, Mark
CORPORATE SOURCE: Department of Medicinal Chemistry and Molecular Pharmacology, School of Pharmacy and Pharmacal Sciences, Purdue University, West Lafayette, IN, 47907, USA
SOURCE: Journal of Medicinal Chemistry (2003), 46(26), 5712-5724
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal

L4 ANSWER 5 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
LANGUAGE: English
OTHER SOURCE(S): CASREACT 140:111315
REFERENCE COUNT: 44 THERE ARE 44 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 6 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB Diamines, substituted by bis(trimethylsilyl)methyl group were prepared either from bis-imination reaction of 1,2-dicarbonyl compds. with bis(trimethylsilyl)methylamine, BSMA (1) followed by reduction or from cyanoethylation of BSMA followed by reduction of the nitrile group. Reaction of R1COCOR2 with two equiv of 1 followed by LiAlH4 reduction gave RNHCHR1CHR2NHR (3a-c, R = (Me3Si)2CH, R1, R2 = H, Me). Cyanoethylation of RNH2 by acrylonitrile gave mono- and bis-cyanoethyl derivs., RNHCH2CH2CN (5) and RN(CH2CH2CN)2 (6), which were reduced to RNH(CH2)3NH2 (7) and RN[(CH2)3NH2]2 (8), resp. The latter is the N-[bis(trimethylsilyl)methyl]-substituted analog of natural polyamine, caldine.
ACCESSION NUMBER: 2003:827493 CAPLUS
DOCUMENT NUMBER: **140:59696**
TITLE: Syntheses of novel N-[bis(trimethylsilyl)methyl]-1,2- and 1,3-diamines
AUTHOR(S): Picard, Jean-Paul; Fortis, Frederic; Grelier, Stephane
CORPORATE SOURCE: Laboratoire de Chimie Organique et Organometallique, UMR 5802 CNRS, Universite Bordeaux 1, Talence, 33405, Fr.
SOURCE: Journal of Organometallic Chemistry (2003), 686(1-2), 306-312
CODEN: JORCAI; ISSN: 0022-328X
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 140:59696
REFERENCE COUNT: 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 7 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
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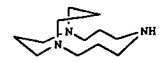
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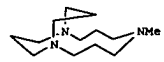
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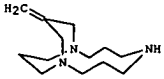
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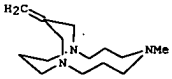

XVI

AB The multistep syntheses of several bicyclic triamines are described, all of which have an imbedded 1,5,9-triazacyclododecane ring. In 1,5,9-triazabicyclo[7.3.3]pentadecanes 12, 13, 15, and 16, (XII, XIII, XV, and XVI, resp.) two nitrogens are bridged by three carbons. The monoprotonated forms of these triamines are highly stabilized by a hydrogen-bonded network involving the bridge and both bridgehead nitrogens, producing a difference of more than 8 pKa units in acidities of their monoprotonated and diprotonated forms. The one- and zero-carbon bridges in 1,5,9-triazabicyclo[9.1.1]tridecane and 7-methyl-1,5,9-triazabicyclo[5.5.0]dodecane do not enhance the stabilities of their monoprotonated forms. X-ray crystal structures and computational studies of 12·HI and 16·HI reveal similar, but somewhat weaker, hydrogen-bonded networks, relative to 15·HI. The activation free energies for conformational inversion of 13·HI (14.4 ± 0.2 kcal/mol), 16·HI (15.0 ± 0.1 kcal/mol) and 16 (8.8 ± 0.3 kcal/mol) were measured by variable-temperature 1H and 13C NMR spectroscopy. These exptl. barriers give an estimate of 6.2 kcal/mol for the strength of the bifurcated hydrogen bond between the bridge nitrogen and cavity proton in 16·HI. Computational studies support the hypothesis that N-inversion occurs in an open conformation, leading to an estimate of 10.32 kcal/mol for the enthalpy of the bifurcated hydrogen bond in 16·HI in the gas phase. Safety: explosion hazard; air must be completely replaced by H2 in Parr apparatus before hydrogenation of bis(2-cyanoethyl)benzylamine.

ACCESSION NUMBER: 2003:727508 CAPLUS
DOCUMENT NUMBER: **139:350441**
TITLE: Syntheses, Conformations, and Basicities of Bicyclic Triamines
AUTHOR(S): Bell, Thomas W.; Choi, Heung-Jin; Harte, William; Drew, Michael G. B.
CORPORATE SOURCE: Department of Chemistry, University of Nevada, Reno, NV, 89557-0020, USA
SOURCE: Journal of the American Chemical Society (2003), 125(40), 12196-12210
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 139:350441
REFERENCE COUNT: 94 THERE ARE 94 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 8 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB Primary amines were prepared by hydrogenation of nitriles in the presence of catalysts containing Co and optionally Ni as well as ≥1 doping metal on a particulate substrate, whereby the Co and optional Ni have an avg. particle size of 3-30 nM. Thus, dimethylaminopropionitrile was hydrogenated in the presence of a suspension catalyst [prepared from Co(NO3)2, Ni(NO3)2, and Y(NO3)3 and aluminosilicate powder] at 80° in the presence of NH3 and 80 bar H2 to give dimethylaminopropylamine in 98.4% selectivity.

ACCESSION NUMBER: 2003:332011 CAPLUS
DOCUMENT NUMBER: **138:337704**
TITLE: Preparation of primary amines via reduction of nitriles in the presence of supported cobalt catalysts containing dopants and optionally containing nickel.
INVENTOR(S): Ansmann, Andreas; Benisch, Christoph
PATENT ASSIGNEE(S): BASF AG, Germany
SOURCE: Ger. Offen., 14 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10152135	A1	20030430	DE 2001-10152135	20011023
US 2003120115	A1	20030626	US 2002-271977	20021017
US 6790996	B2	20040914		
EP 1306365	A2	20030502	EP 2002-23640	20021021
EP 1306365	A3	20031015		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK				
JP 2003192647	A2	20030709	JP 2002-307884	20021023
PRIORITY APPLN. INFO.:			DE 2001-10152135 A	20011023

OTHER SOURCE(S): CASREACT 138:337704; MARPAT 138:337704

L4 ANSWER 9 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB The total synthesis and the revised structural assignment of petrobactin, a siderophore isolated from the marine bacterium Marinobacter hydrocarbonoclasticus, is reported. The key step in the synthesis involved condensation of N1-(2,3-dibenzoyloxybenzoyl)-N4-benzylspermidine with 1,3-di-(p-nitrophenyl)-2-tert-Bu citrate. Proton NMR spectra of the synthesized product compared with those reported for the natural product revealed that the compound did not contain 2,3-dihydroxybenzoyl moieties as published; instead, the splitting pattern suggested 3,4-dihydroxybenzoyl fragments. The 3,4-dihydroxybenzoyl analog was accessed via a similar route; the proton and carbon-13 NMR spectra of this compound were consistent with those reported for natural petrobactin.

ACCESSION NUMBER: 2003:163281 CAPLUS
DOCUMENT NUMBER: **139:53282**
TITLE: Total synthesis and structure revision of petrobactin
AUTHOR(S): Bergeron, Raymond J.; Huang, Guangfei; Smith, Richard E.; Bharti, Neelam; McManis, James S.; Butler, Alison
CORPORATE SOURCE: Department of Medicinal Chemistry, University of Florida, Gainesville, FL, 32610, USA
SOURCE: Tetrahedron (2003), 59(11), 2007-2014
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 139:53282
REFERENCE COUNT: 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

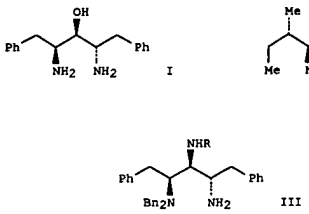
L4 ANSWER 10 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Analogs of 1-[[(4R)-4,5-dihydro-2-phenyl-1H-imidazol-4-yl]methyl]-4-phenylpiperazine (FAUC-179) were prepared Investigation of conformationally restricted benzamide bioisosteres led to a chiral phenyltetrahydropyrimidine derivative, (-)-(6R)-3,4,5,6-tetrahydro-2-phenyl-6-[(4-phenyl-1-piperazinyl)methyl]pyrimidine (FAUC 312) displaying strong and highly selective dopamine D4 receptor binding (Kihigh=1.5 nM). Mitogenesis expts. indicated 83% ligand efficacy when compared to the unselective agonist quinpirole. The target compds. were synthesized in enantiopure form starting from asparagine. Compds. thus prepared included (+)-(6S)-3,4,5,6-tetrahydro-2-phenyl-6-[(4-phenyl-1-piperazinyl)methyl]pyrimidine, (-)-(6R)-3,4,5,6-tetrahydro-2-phenyl-6-[(4-phenyl-1-piperazinyl)methyl]pyrimidine, (5R)-4,5,6,7-tetrahydro-2-phenyl-5-[(4-phenyl-1-piperazinyl)methyl]-1H-1,3-diazepine, (5S)-4,5,6,7-tetrahydro-2-phenyl-5-[(4-phenyl-1-piperazinyl)methyl]-1H-1,3-diazepine, (6S)-5,6-dihydro-2-phenyl-6-[(4-phenyl-1-piperazinyl)methyl]-4(3H)-pyrimidinone and (6R)-5,6-dihydro-2-phenyl-6-[(4-phenyl-1-piperazinyl)methyl]-4(3H)-pyrimidinone. These compds. were analogs of 1-[[(4R)-4,5-dihydro-2-phenyl-1H-imidazol-4-yl]methyl]-4-phenylpiperazine (FAUC-179).

ACCESSION NUMBER: 2003:162655 CAPLUS
DOCUMENT NUMBER: **139:117402**
TITLE: Cyclic Amidines as Benzamide Bioisosteres: EPC Synthesis and SAR Studies Leading to the Selective Dopamine D4 Receptor Agonist FAUC 312
AUTHOR(S): Einsiedel, Jurgen; Hubner, Harald; Gmeiner, Peter
CORPORATE SOURCE: Emil Fischer Center, Department of Medicinal Chemistry, Friedrich-Alexander University, Erlangen, D-91052, Germany
SOURCE: Bioorganic & Medicinal Chemistry Letters (2003), 13(5), 851-854
CODEN: BMCLE8; ISSN: 0960-894X
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 139:117402
REFERENCE COUNT: 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 11 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

GI



AB Nonracemic pseudo-C2-sym. diamino alc. and triamines are prepared stereoselectively from amino acid-derived aldehydes as potential fragments for HIV protease inhibitors. Addition of organolithium reagents to siloxy nitriles derived from L-phenylalaninal and L-isoleucinal, followed by in situ reduction of the intermediate imines and transfer hydrogenation and deprotection of N-benzyl protecting groups under microwave irradiation, led to 1,3-diamino alcs. such as I and II in moderate to good yields. Nonracemic pseudo-C2-sym. triamine III (R = 4-MeOC6H4) is prepared using a scandium triflate-catalyzed nitro-Mannich addition of the silylnitronate PhCH2CH:N+(O-)OSiMe3 derived from 2-phenyl-1-nitroethane to (S)-PhCH2CH(NBn2)CH:N-4-C6H4OMe, derived from L-phenylalaninal; reduction of the nitro group with sodium borohydride and nickel (II) chloride, and separation of diastereomers yields III (R = 4-MeOC6H4).

ACCESSION NUMBER: 2003:50024 CAPLUS
DOCUMENT NUMBER: **138:237779**
TITLE: Concise and Stereocontrolled Synthesis of Pseudo-C2-symmetric Diamino Alcohols and Triamines for Use in HIV Protease Inhibitors
AUTHOR(S): Bernardi, Luca; Bonini, Bianca F.; Dessole, Gabriella; Fochi, Mariafrancesca; Comes-Franchini, Mauro; Gavioli, Silvia; Ricci, Alfredo; Varchi, Greta
CORPORATE SOURCE: Dipartimento di Chimica Organica "A. Mangini", Facolta di Chimica Industriale, Universita di Bologna, Bologna, 40136, Italy
SOURCE: Journal of Organic Chemistry (2003), 68(4), 1418-1425
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal

L4 ANSWER 11 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:237779
REFERENCE COUNT: 43 THERE ARE 43 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 12 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB The ability to generate RNA mols. that can catalyze complex organic transformations not only facilitates the reconstruction and plausibility of possible prebiotic reaction pathways but is also crucial for elucidating the potential of the application of RNA catalysts in organic syntheses. Iterative RNA selection previously identified a ribozyme that catalyzes the Michael addition of a cysteine thiol to an α,β-unsatd. amide. This reaction is chemical similar to the rate limiting step of the thymidylate synthase reaction, which is the corresponding reaction of a cysteine thiol to the double-bond of the uracil nucleobase. Here we provide a detailed description of the synthesis of the ribozyme substrates and the substrate oligonucleotides used for its characterization and the investigation of the background reaction. We also describe the further characterization of the ribozyme with respect to substrate specificity. We show that the thiol group of the cysteine nucleophile is essential for the reaction to proceed. When substituted for a thiomethyl group, no reaction takes place.

ACCESSION NUMBER: 2002:915648 CAPLUS
DOCUMENT NUMBER: **138:299672**
TITLE: A Ribozyme with Michaelase Activity: Synthesis of the Substrate Precursors
AUTHOR(S): Eisenfuhr, Alexander; Arora, Paramjit S.; Sengle, Gerhard; Takaoka, Leo R.; Nowick, James S.; Famulok, Michael
CORPORATE SOURCE: Kekule-Institut fur Organische Chemie und Biochemie, Rheinische Friedrich-Wilhelms-Universitat Bonn, Bonn, 53121, Germany
SOURCE: Bioorganic & Medicinal Chemistry (2003), 11(2), 235-249
CODEN: BMECEP; ISSN: 0968-0896
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:299672
REFERENCE COUNT: 49 THERE ARE 49 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 13 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB A series of diamine and polyamine deriva., either free amines or salts (HCl or TFA), of aspartic and glutamic acid were prepared in excellent yields using Rink Amide solid-phase synthesis. The asparagine and glutamine derivs. were all evaluated for their ability to inhibit Tat-TAR binding using a FIGS (fusion induced gene stimulating) cellular assay, with the polyamine derivs. exhibiting the most promising binding activity.

ACCESSION NUMBER: 2002:915629 CAPLUS
DOCUMENT NUMBER: **138:304514**
TITLE: Solid-Phase synthesis of diamine and polyamine amino acid derivatives as HIV-1 tat-TAR binding inhibitors
AUTHOR(S): Jimenez Bueno, G.; Klimkait, T.; Gilbert, I. H.; Simons, C.
CORPORATE SOURCE: Welsh School of Pharmacy, Cardiff University, Cardiff, CF10 3XF, UK
SOURCE: Bioorganic & Medicinal Chemistry (2003), 11(1), 87-94
CODEN: BMECEP; ISSN: 0968-0896
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:304514
REFERENCE COUNT: 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 14 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Bisphosphonates (BPs) are pyrophosphate analogs in which the oxygen in P-O-P has been replaced by a carbon, resulting in a metabolically stable P-C-P structure. Pamidronate (1b, Novartis), a second-generation BP, was the starting point for extensive SAR studies. Small changes of the structure of pamidronate lead to marked improvements of the inhibition of osteoclastic resorption potency. Alendronate (1c, MSD), with an extra methylene group in the N-alkyl chain, and olpadronate (1h, Gador), the N,N-di-Me analog, are about 10 times more potent than pamidronate. Extending one of the N-Me groups of olpadronate to a pentyl substituent leads to ibandronate (1k, Roche, Boehringer-Mannheim), which is the most potent close analog of pamidronate. Even slightly better antiresorptive potency is achieved with derivs. having a Ph group linked via a short aliphatic tether of three to four atoms to nitrogen, the second substituent being preferentially a Me group (e.g., 4g, 4j, 5d, or 5r). The most potent BPs are found in the series containing a heteroarom. moiety (with at least one nitrogen atom), which is linked via a single methylene group to the geminal bisphosphonate unit. Zoledronic acid (6i), the most potent derivative, has an ED50 of 0.07 mg/kg in the TPTX in vivo assay after s.c. administration. It not only shows by far the highest therapeutic ratio when comparing resorption inhibition with undesired inhibition of bone mineralization but also exhibits superior renal tolerability. Zoledronic acid (6i) has thus been selected for clin. development under the registered trade name Zometa. The results of the clin. trials indicate that low doses are both efficacious and safe for the treatment of tumor-induced hypercalcemia, Paget's disease of bone, osteolytic metastases, and postmenopausal osteoporosis.

ACCESSION NUMBER: 2002:539062 CAPLUS
DOCUMENT NUMBER: **137:226194**
TITLE: Highly Potent Geminal Bisphosphonates. From Pamidronate Disodium (Aredia) to Zoledronic Acid (Zometa)
AUTHOR(S): Widler, Leo; Jaeggi, Knut A.; Glatt, Markus; Mueller, Klaus; Bachmann, Rolf; Bisping, Michael; Born, Anne-Ruth; Cortesi, Reto; Guiglia, Gabriela; Jeker, Heidi; Klein, Remy; Ramseier, Ueli; Schmid, Johann; Schreiber, Gerard; Seltenmeyer, Yves; Green, Jonathan R.
CORPORATE SOURCE: Arthritis and Bone Metabolism Therapeutic Area, Novartis Pharma Research, Basel, CH-4002, Switz.
SOURCE: Journal of Medicinal Chemistry (2002), 45(17), 3721-3738
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 137:226194
REFERENCE COUNT: 88 THERE ARE 88 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 15 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB A series of dendritic polyamines-(imide-DNA-intercalators) conjugates with different connectivity in their basic chain were synthesized and evaluated as antitumor compds. Although their antiproliferative activity against HT-29 was not significant, conjugates 13 and 16 showed a promising profile as inhibitors of Lck.

ACCESSION NUMBER: 2002:532512 CAPLUS
DOCUMENT NUMBER: **138:214841**
TITLE: Synthesis and antitumour activity of new dendritic polyamines-(imide-DNA-intercalator) conjugates: potent Lck inhibitors
AUTHOR(S): Brana, Miguel F.; Dominguez, Gema; Saez, Beatriz; Romerdahl, Cynthia; Robinson, Simmon; Barlozzari, Teresa
CORPORATE SOURCE: Facultad de Ciencias Experimentales y Tecnicas, Departamento de Quimica Organica y Farmaceutica, Universidad San Pablo-CEU, Boadilla del Monte, Madrid, 28668, Spain
SOURCE: European Journal of Medicinal Chemistry (2002), 37(7), 541-551
CODEN: EJMCA5; ISSN: 0223-5234
PUBLISHER: Editions Scientifiques et Medicales Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:214841
REFERENCE COUNT: 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 16 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

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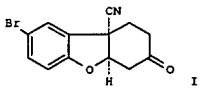

I

AB A regiocontrolled synthesis of the macrocyclic polyamine alkaloid (±)-lunarine is described. The key steps involve the preparation of the differentially functionalized cis-3-oxo-8-bromo-9b-cyano-1,2,3,4,4a,9b-hexahydrobenzofuranyl tricyclic scaffold I which, following further elaboration, is coupled to the selectively protected acrylamidospermidine via a Heck coupling reaction to give a pre-cyclized lunarine derivative.

ACCESSION NUMBER: 2002:265151 CAPLUS
DOCUMENT NUMBER: **137:140669**
TITLE: Regiocontrolled synthesis of the macrocyclic polyamine alkaloid (±)-lunarine, a time-dependent inhibitor of trypanothione reductase
AUTHOR(S): Hamilton, Chris J.; Fairlamb, Alan H.; Eggleston, Ian M.
CORPORATE SOURCE: Division of Biological Chemistry and Molecular Microbiology, School of Life Sciences, University of Dundee, Dundee, DD1 4HN, UK
SOURCE: Journal of the Chemical Society, Perkin Transactions 1 (2002), (8), 1115-1123
CODEN: JCSPCE; ISSN: 1472-7781
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 137:140669
REFERENCE COUNT: 47 THERE ARE 47 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 17 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Dendritic imides RCH2CH2{(CH2)3[(CH2)3N(CH2CH2NH2)2]2}2 [I, R = 1,8-naphthalimido, 3-nitro-1,8-naphthalimido, 3-amino-1,8-naphthalimido, diphenylmaleimido] were synthesized and evaluated as antitumor compds. I [R = 1,8-naphthalimido, diphenylmaleimido] showing a promising profile as inhibitors of lck but their antiproliferative activity against HT-29 was not so relevant.

ACCESSION NUMBER: 2001:872196 CAPLUS
DOCUMENT NUMBER: **136:309833**
TITLE: Synthesis of antitumor dendritic imides
AUTHOR(S): Brana, Miguel F.; Dominguez, Gema; Saez, Beatriz; Romerdahl, Cynthia; Robinson, Simmon; Barlozzari, Teresa
CORPORATE SOURCE: Department of Organic and Pharmaceutical Chemistry, University San Pablo-CEU, Madrid, 28668, Spain
SOURCE: Bioorganic & Medicinal Chemistry Letters (2001), 11(23), 3027-3029
CODEN: BMCLE8; ISSN: 0960-894X
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 136:309833
REFERENCE COUNT: 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 18 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB The P2 transporter is a nucleoside transporter which is unique to the protozoan parasite Trypanosoma brucei, the causative organism of Human African Trypanosomiasis. The transporter has been shown to bind some structural motifs not recognized by other transporters. In this paper we describe the use of the melamine motif, a substrate of the P2 transporter, as a potential tool to selectively deliver polyamine analogs to the parasites. The synthesis of a number of polyamine analogs attached to a variety of melamine analogs is described. Many of the compds. were shown to competitively inhibit uptake of adenosine, indicating that they are recognized by the transporter. Some of the compds. showed good in vitro activity against the parasites.

ACCESSION NUMBER: 2001:680366 CAPLUS
DOCUMENT NUMBER: **135:366327**
TITLE: Synthesis and Biological Evaluation of s-Triazine Substituted Polyamines as Potential New Anti-Trypanosomal Drugs
AUTHOR(S): Klenke, Burkhard; Stewart, Mhairi; Barrett, Michael P.; Brun, Reto; Gilbert, Ian H.
CORPORATE SOURCE: Welsh School of Pharmacy, Cardiff University, Cardiff, CF10 3XF, UK
SOURCE: Journal of Medicinal Chemistry (2001), 44(21), 3440-3452
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 135:366327
REFERENCE COUNT: 51 THERE ARE 51 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 19 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Synthesis of poly(propyleneimine) dendrimer was studied. By optimizing reaction conditions, such as increasing reaction temperature and concentration of reactant, introducing a little acetic acid as catalyst and choosing ammonia/methanol as solvent, a preparative route was developed in which the poly(propyleneimine) dendrimer with relatively perfect structure was obtained and the side reactions were suppressed to a considerable degree or even eliminated. IR spectrum showed that 0.5 generation poly(propyleneimine) dendrimer was obtained by reacting completely under optimum conditions, which was in favor of the growth of structure. Titration showed that 1.0 generation poly(propyleneimine) dendrimer obtained in ammonia/methanol contained 85 percent primary amine, which was better than that obtained in methanol. 1H NMR and elementary anal. also showed that structures of low generation poly(propyleneimine) dendrimer were perfect and in accord with the expected mol. structure.

ACCESSION NUMBER: 2001:631049 CAPLUS
DOCUMENT NUMBER: **136:167074**
TITLE: Synthesis and characterization of low generation poly(propyleneimine) dendrimer
AUTHOR(S): Wang, Gang; Luo, Yun-jun; Tan, Hui-min
CORPORATE SOURCE: School of Chemical Engineering and Materials Science, Beijing Institute of Technology, Beijing, 100081, Peop. Rep. China
SOURCE: Jingxi Huagong (2001), 18(7), 414-416, 420
CODEN: JIHUFJ; ISSN: 1003-5214
PUBLISHER: Jingxi Huagong Bianjibu
DOCUMENT TYPE: Journal
LANGUAGE: Chinese
OTHER SOURCE(S): CASREACT 136:167074

L4 ANSWER 20 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

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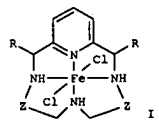

I

AB Iron(III) complexes of macrocyclic tetraaza ligands I+(BF4-) [R = Me, Z = (CH2)2 (Fe(III)-1); R = H, Z = (CH2)2 (Fe(III)-2); R = H, Z = o-phenylene (Fe(III)-3)] were prepared and studied for their putative catalase-like properties under physiol. conditions: i.e., in aqueous solution at pH = 7.0-7.4 and at micromolar concns. of the catalyst and H2O2. Complex Fe(III)-1, originally studied by Busch et al. as a catalase model, at pH = 4.6, only degrades H2O2 to O2 as a minor reaction at this pH (< 1% O2 yield). Expts. with the analogous complex Fe(III)-2 at pH = 7.2 found that no O2 was formed under physiol. conditions, although H2O2 was decomposed to an extent of >50%. Complex Fe(III)-3 produced O2 on reaction with H2O2, but only in stoichiometric amts. Thus, the decomposition of H2O2 by these Fe(III) complexes cannot reasonably be described as a catalase-like activity.

ACCESSION NUMBER: 2001:616493 CAPLUS
DOCUMENT NUMBER: **135:326608**
TITLE: Reactions of tetraazamacrocyclic FeIII complexes with hydrogen peroxide - putative catalase mimics?
AUTHOR(S): Autzen, Sabrina; Korth, Hans-Gert; De Groot, Herbert; Sustmann, Reiner
CORPORATE SOURCE: Institut fur Organische Chemie, Universitat Essen, Essen, 45117, Germany
SOURCE: European Journal of Organic Chemistry (2001), (16), 3119-3125
CODEN: EJOCFK; ISSN: 1434-193X
PUBLISHER: Wiley-VCH Verlag GmbH
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 135:326608
REFERENCE COUNT: 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 21 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB Several polyamine derivs. were synthesized in order to produce novel antagonists of muscular nicotinic acetylcholine receptors. Their affinities were compared with those of philanthotoxin PhTX-343.

ACCESSION NUMBER: 2001:516946 CAPLUS
DOCUMENT NUMBER: **135:288945**
TITLE: Analogues of polyamine alkaloids and their synthetic advantages
AUTHOR(S): Li, Yi; Popaj, Kasim; Lochner, Martin; Geneste, Herve;
Budriesi, Roberta; Chiarini, Alberto; Melchiorre, Carlo; Hesse, Manfred
CORPORATE SOURCE: Organisch-chemisches Institut, Universitat Zurich, Zurich, CH-8057, Switz.
SOURCE: Farmaco (2001), 56(1-2), 127-131
CODEN: FRMCE8; ISSN: 0014-827X
PUBLISHER: Elsevier Science S.A.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 135:288945
REFERENCE COUNT: 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 22 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB Primary amines are prepared by catalytic hydrogenation of nitriles in the presence of W-containing Raney Ni catalysts. Ni-Al-W alloy (43:56:1) was heated in aqueous NaOH at 95-100° for 2 h to give a catalyst, which was used in hydrogenation of
3-[N-(2-hydroxyethyl)-N-methylamino]propionitrile
in the presence of aqueous NH3 at 65° under 2.0 MPa for 2.0 h to give 97% N-(2-hydroxyethyl)-N-methyl-1,3-propanediamine.

ACCESSION NUMBER: 2001:496310 CAPLUS
DOCUMENT NUMBER: **135:92364**
TITLE: Preparation of primary amines and catalysts for reduction of nitriles
INVENTOR(S): Kikuchi, Ryuji; Nagai, Naofumi; Arakawa, Tatsuya
PATENT ASSIGNEE(S): Kawaken Fine Chemicals Co., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2001187766	A2	20010710	JP 2000-123106	20000424
PRIORITY APPLN. INFO.:			JP 1999-301544 A	19991022

OTHER SOURCE(S): CASREACT 135:92364

L4 ANSWER 23 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
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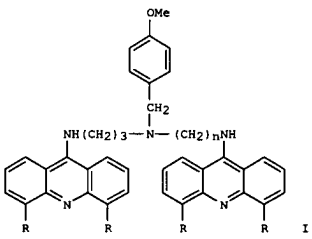

AB Methods for the synthesis of N1,N8-bis(9-acridinyl)-N4-(4-hydroxybenzyl)spermidine and N1,N7-(hydroxybenzyl)bis(3-aminopropyl)amine were investigated. Thus, monocyanoethylation of 4-methoxybenzylamine followed by treatment with 4-chlorobutyronitrile gave the dinitrile N-(2-cyanoethyl)-N-(3-cyanopropyl)-4-methoxybenzylamine. Subsequent in situ reduction with lithium aluminum hydride gave the corresponding diamine.
Biscyanoethylation of 4-methoxybenzylamine with 2 mol of acrylonitrile followed by reduction yielded the diamine N,N-bis(3-aminopropyl)-4-methoxybenzylamine. Both diamines reacted smoothly with 9-methoxyacridine
to give the bis(9-acridinyl) compds. I (n = 3, 4; R = H) but with 4,5-dimethyl-9-methoxyacridine, the bis compound I (n = 4, R = Me) was produced in only low yields. Demethylation of the dinitriles by a variety
of approaches all failed to give the corresponding hydroxybenzyl derivs. These studies yielded useful methylated tyrosine derivs. which could also be iodinated. This study has been useful for elucidating chemical methods
needed for the synthesis of the desired tyrosine-based bis acridine compound
and for alerting us to the need to synthesize a more labile protected tyrosine intermediate which will be easily deprotected to afford the desired tyrosine-based bis acridine compound

ACCESSION NUMBER: 2001:357493 CAPLUS
DOCUMENT NUMBER: **136:151064**
TITLE: Synthesis of acridine-based DNA bis-intercalating agents
AUTHOR(S): Moloney, Gerard P.; Kelly, David P.; Mack, P.
CORPORATE SOURCE: The Austin Research Institute, Victoria, 3084, Australia
SOURCE: Molecules [online computer file] (2001), 6(3), 230-243
CODEN: MOLEFW; ISSN: 1420-3049
URL: http://www.mdpi.org/molecules/papers/60300230.pdf
PUBLISHER: Molecular Diversity Preservation International

L4 ANSWER 23 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
DOCUMENT TYPE: Journal; (online computer file)
LANGUAGE: English
OTHER SOURCE(S): CASREACT 136:151064
REFERENCE COUNT: 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 24 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Ni(II) complexes with the aliphatic tripodal tetraamine ligands N(CH2CH2NH2)3 (tren, 1), N[(CH2CH2CH2NH2)(CH2CH2NH2)2] (baep, 2), N[(CH2CH2CH2NH2)2(CH2CH2NH2)] (abap, 3), and N(CH2CH2CH2NH2)3 (trpn, 4) are reported. The tripodal tetradentate N4 ligands 1-4 react with Ni(NO3)2·6H2O in MeCN or MeOH to give the blue Ni(II) complexes [Ni(1)(η1-NO3)2] (5a), [Ni(2)(η1-NO3)2] (5b), [Ni(3)(η2-NO3)]NO3·MeCN (5c·MeCN), and [Ni(4)(η2-NO3)]NO3·MeOH (5d·MeOH). With NiCl2·6H2O, [Ni(1)Cl(H2O)]Cl·H2O (6a), [Ni(2)(μ-Cl)2Ni(2)]Cl2·2MeOH (6b·2MeOH), [Ni(3)Cl(H2O)]Cl (6c), and [Ni(4)(H2O)2]Cl2·H2O (6d·H2O) were obtained. The mol. structures of 5a-d and 6b-d were determined by x-ray diffraction anal. and they are compared with the mol. structure of the previously characterized complex 6a. Complexes 5a-d and 6b-d exhibit octahedrally coordinated Ni atoms. The tripodal ligands occupy four of the six coordination sites in a pseudo-facial manner. Complexes of the unsym. 2 and 3 possess both five- and six-membered chelate rings. The extension of the ligand arms in 1-4 leads to a systematic variation in the geometric and UV/visible spectroscopic properties of the complexes depending on the size of the chelate rings formed by the ligands.

ACCESSION NUMBER: 2001:335178 CAPLUS
DOCUMENT NUMBER: **135:115961**
TITLE: Cis-octahedral nickel(II) complexes with symmetric and unsymmetric tripodal tetraamine ligands
AUTHOR(S): Ochs, Christian; Hahn, F. Ekkehardt; Lugger, Thomas
CORPORATE SOURCE: Institut fur Anorganische und Analytische Chemie der Freien Universitat Berlin, Berlin, 14195, Germany
SOURCE: European Journal of Inorganic Chemistry (2001), (5), 1279-1285
CODEN: EJICFO; ISSN: 1434-1948
PUBLISHER: Wiley-VCH Verlag GmbH
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 135:115961
REFERENCE COUNT: 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 25 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Nitriles NCCH2CH2NX-R-NXCH2CH2CN [R = (CH2)n, 1,2-cyclohexyl, 1,4-cyclohexyl, CHMeCH2, X = Boc, n = 4, 7, 9, 12; R = 1,2-Ph, 1,3-Ph, 1,4-Ph, X = H] are reduced to primary amines in the presence of N-tert-butoxycarbonyl (Boc) groups. E.g., NCCH2CH2N(Boc)(CH2)4N(Boc)CH2CH2CN in absolute ethanol and THF was stirred with a mixture of Pd/C and Raney nickel and sodium hydroxide; the mixture was shaken for 8 h under 45 psi of hydrogen and worked up to give H2N(CH2)3N(Boc)(CH2)4N(Boc)(CH2)3NH2 in 84% yield. The reduction can be carried out under atmospheric H2 pressure using com. avail. catalysts. Both Boc groups and aromatic moieties present in the starting material are well tolerated under the mild optimized conditions.

ACCESSION NUMBER: 2001:156286 CAPLUS
DOCUMENT NUMBER: **134:326005**
TITLE: Nitrile reduction in the presence of Boc-protected amino groups by catalytic hydrogenation over palladium-activated Raney-nickel
AUTHOR(S): Klenke, Burkhard; Gilbert, Ian H.
CORPORATE SOURCE: Welsh School of Pharmacy, Cardiff University, Cardiff, CF10 3XF, UK
SOURCE: Journal of Organic Chemistry (2001), 66(7), 2480-2483
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:326005
REFERENCE COUNT: 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 26 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Hydrogenation of 3-(dimethylamino)propionitrile over palladium catalysts was studied. Besides the expected amines, N,N,N',N'-tetramethylpropane-1,3-diamine, N,N-dimethyl-N'-propyl-propane-1,3-diamine, and N,N-bis[3-(dimethylamino)propyl]propylamine were found. Reaction pathways of their formation were discussed. Effects of reaction conditions, type of catalyst, and addition of ammonia or an amine into the charge on the hydrogenation selectivity were studied.

ACCESSION NUMBER: 2001:61467 CAPLUS
DOCUMENT NUMBER: **134:266016**
TITLE: Hydrogenation of 3-(dimethylamino)propionitrile over palladium catalysts
AUTHOR(S): Krupka, Jiri; Pasek, Josef; Navratilova, Marketa
CORPORATE SOURCE: Department of Organic Technology, Institute of Chemical Technology, Prague, Prague, 166 28/6, Czech Rep.
SOURCE: Collection of Czechoslovak Chemical Communications (2000), 65(11), 1805-1819
CODEN: CCCCAK; ISSN: 0010-0765
PUBLISHER: Institute of Organic Chemistry and Biochemistry, Academy of Sciences of the Czech Republic
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:266016
REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 27 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB A lipid-polypeptide conjugate (lipo-polypeptide) was obtained by the ring-opening polycondensation of N-α-Z-L-lysine N-carboxyanhydride (NCA) using 3-aminopropyl dioctadecylamine as initiator and subsequent deprotection. Maltose lactone was coupled with the lipo-polypeptide to give novel amphiphiles which carried many maltoamide residues as pendent groups. The sugar group-carrying amphiphiles incorporated in phospholipid liposomes were recognized by a lectin from Canavalia ensiformis (Con A), which was proven by the increase in turbidity of the liposome suspension after mixing with the lectin. The recognition was largely affected by the d.p. of lysine residues and the surface d. of the amphiphile in the liposomes. The association constant (Kass) of Con A with maltoamide residues on the liposome was much larger than those for small mol. weight sugars due to the "cluster effect".

ACCESSION NUMBER: 2000:909857 CAPLUS
DOCUMENT NUMBER: **134:204109**
TITLE: Recognition of Novel Lipopolypeptides with Many Pendent Sugar Residues by Lectin
AUTHOR(S): Kitano, Hiromi; Sumi, Yusuke; Tagawa, Koji
CORPORATE SOURCE: Department of Chemical and Biochemical Engineering, Toyama University, Toyama, 930-8555, Japan
SOURCE: Bioconjugate Chemistry (2001), 12(1), 56-61
CODEN: BCCHES; ISSN: 1043-1802
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:204109
REFERENCE COUNT: 34 THERE ARE 34 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 28 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Pyrrolidyl polyamines are conformationally restricted, chiral analogs of linear spermine elaborated by the addition of aminopropyl chains to yield branched diastereomers. It is demonstrated that in concns. as low as 0.01 mM, these compds. remarkably stabilize DNA duplexes and triplexes through strong electrostatic interactions. The synthesized compds. are potential dendrons with a chiral pyrrolidine core, and such mols. may have potential as DNA delivery and transfection agents.

ACCESSION NUMBER: 2000:887091 CAPLUS
DOCUMENT NUMBER: 134:189584
TITLE: Pyrrolidyl Polyamines: Branched, Chiral Polyamine Analogues That Stabilize DNA Duplexes and Triplexes
AUTHOR(S): Nagamani, Dendukuri; Ganesh, Krishna N.
CORPORATE SOURCE: Division of Organic Chemistry (Synthesis), National Chemical Laboratory, Pune, 411008, India
SOURCE: Organic Letters (2001), 3(1), 103-106
CODEN: ORLEF7; ISSN: 1523-7060
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:189584
REFERENCE COUNT: 31 THERE ARE 31 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 29 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 29 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

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* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Starting from aspartic acid, the authors synthesized lactam-bridged β- and γ-amino acid equivalent Using the 1,4-bis electrophile (PhCH2)2NCH(CH2OSO2Me)CH2CH2OSO2Me as a central intermediate, the 4- and 5-aminopiperidin-2-ones I and II (R = H, Me3COCO, PhCH2) were prepared by regioselective functionalization and subsequent lactamization. Diastereoselective C-alkylation was performed after N-protection of the lactam functionality when exclusive trans configuration resulting in the formation of III (R = Me3COCO, CH2Ph; R1 = Me, PhCH2, F) was observed in the 4-amino series. On the other hand, cis selectivity was typical for the alkylations of the 5-amino lactams III (R = Boc; R1 = Me, PhCH2). To investigate the ability of the lactam building blocks to induce reverse-turn structures by intramol. hydrogen bonding, the model peptidomimetics IV and V representing Homo-Freidinger lactams were prepared from I and II (R = H), resp. Conformational analyses in dilute solution (1 mM) by IR and NMR spectroscopy at room temperature clearly indicated that the 4-aminopiperidin-2-one derivative IV predominantly adopts a reverse-turn structure stabilized by a CO-HN hydrogen bond in an 11-membered ring. VT NMR expts. showed a substantial temperature dependency of the terminal NH when ΔδNH/ΔT = -6.5 indicated that the amount of intramol. hydrogen bonding is higher at low temperature An application in the field of medicinal chemical was demonstrated. Thus, starting from a Homo-Freidinger lactam and its enantiomer, the authors synthesized the peptidomimetics (2S,4S)-VI and (2S,4R)-VI and investigated them as lactam-bridged analogs of the dopamine receptor modulating peptide Pro-Leu-Gly-NH2 (PLG). Both test compds. turned out to enhance significantly the agonist binding of dopamine D2 receptors, when the isomer 15c revealed a potency comparable to the genuine ligand PLG.

ACCESSION NUMBER: 2000:692927 CAPLUS
DOCUMENT NUMBER: 134:17379
TITLE: Enantiopure 4- and 5-aminopiperidin-2-ones: regiocontrolled synthesis and conformational characterization as bioactive β-turn mimetics
AUTHOR(S): Weber, Klaus; Ohnmacht, Ursula; Gmeiner, Peter
CORPORATE SOURCE: Department of Medicinal Chemistry Emil Fischer Center, Friedrich-Alexander University, Erlangen, D-91052, Germany
SOURCE: Journal of Organic Chemistry (2000), 65(22), 7406-7416
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:17379
REFERENCE COUNT: 46 THERE ARE 46 CITED REFERENCES AVAILABLE FOR THIS

L4 ANSWER 30 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB The synthesis of 1,8-bis[(4-methylphenyl)sulfonyl]-4,11-bis[(trifluoromethyl)sulfonyl]-1,4,8,11-tetraazacyclotetradecane and 1,8-bis[(4-methylphenyl)sulfonyl]-5,12-bis[(2-nitrophenyl)sulfonyl]-1,4,8,11-tetraazacyclotetradecane was reported. This synthesis was designed in such a way that the products and some intermediates could be selectively deprotected providing versatility both during the synthesis and for the utility of the final product. Thus, it should be possible to form cryptand mols. with trans bridges with good selectivity.

ACCESSION NUMBER: 2000:443512 CAPLUS
DOCUMENT NUMBER: 133:252403
TITLE: Total Synthesis of Saturated and Unsaturated di-trans-N-substituted Cyclam-based Macrocycles Through a Versatile Intermediate
AUTHOR(S): Barros, M. T.; Sineriz, F.
CORPORATE SOURCE: Departamento de Quimica, Faculdade de Ciencias e Tecnologia da Universidade Nova de Lisboa, Monte de Caparica, 2825-114, Port.
SOURCE: Tetrahedron (2000), 56(27), 4759-4764
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 133:252403
REFERENCE COUNT: 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 31 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB The universal template approach to drug design foresees that a polyamine can be modified in such a way to recognize any neurotransmitter receptor. Thus, hybrids of polymethylene tetraamines and philanthotoxins, exemplified by methoctramine and PhTX-343, resp., were synthesized to produce novel inhibitors of muscular nicotinic acetylcholine receptors. Polyamines were synthesized and their biol. profiles were evaluated at frog rectus abdominis muscle nicotinic receptors and guinea pig left atria (M2) and ileum longitudinal muscle (M3) muscarinic acetylcholine receptors. All of the compds., like prototypes methoctramine and PhTX-343, were noncompetitive antagonists of nicotinic receptors while being, like methoctramine, competitive antagonists at muscarinic M2 and M3 receptor subtypes. Interestingly, polyamines bearing a low number of methylenes between the nitrogen atoms displayed a biol. profile similar to that of PhTX-343: a noncompetitive antagonism at nicotinic receptors in the 7-25 μM range while not showing any antagonism for muscarinic receptors ≤10 μM. Increasing the number of methylenes separating these nitrogen atoms in methoctramine-related tetraamines resulted in a significant improvement in potency at nicotinic receptors. The most potent tetraamine was one bearing a 12 methylene spacer between the nitrogen atoms; it was 12-fold and 250-fold more potent than prototypes methoctramine and PhTX-343, resp. Tetraamines bearing a rather rigid spacer between the nitrogen atoms instead of the very flexible polymethylene chain displayed a profile similar to that of methoctramine at nicotinic receptors, whereas a significant decrease in potency was observed at muscarinic M2 receptors. This finding may have relevance in understanding the mode of interaction with these receptors. Similarly, the constrained analog of methoctramine showed a decrease in potency at nicotinic and muscarinic M2 receptors, revealing that the tricyclic system, which incorporates the 2-methoxybenzylamine moiety of methoctramine, does not represent a good pharmacophore for activity at these sites. A most intriguing finding was the observation that the photolabile tetraamine was more potent than methoctramine at nicotinic receptors and, what is more important, it inhibited a closed state of the receptor.

ACCESSION NUMBER: 1999:757961 CAPLUS
DOCUMENT NUMBER: **132:73226**
TITLE: Design, Synthesis, and Biological Evaluation of Symmetrically and Unsymmetrically Substituted Methoctramine-Related Polyamines as Muscular Nicotinic Receptor Noncompetitive Antagonists
AUTHOR(S): Rosini, Michela; Budriesi, Roberta; Bixel, M. Gabriele; Bolognesi, Maria L.; Chiarini, Alberto; Hucho, Ferdinand; Krogsgaard-Larsen, Povl; Mellor, Ian R.; Minarini, Anna; Tumiatti, Vincenzo; Usherwood, Peter N. R.; Melchiorre, Carlo
CORPORATE SOURCE: Department of Pharmaceutical Sciences, University of Bologna, Bologna, 40126, Italy
SOURCE: Journal of Medicinal Chemistry (1999), 42(25), 5212-5223
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 132:73226

L4 ANSWER 31 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

REFERENCE COUNT: 34 THERE ARE 34 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 32 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

GI

I

AB β-(Benzimidazol-1-yl)propionitrile (I) was prepared in 91% yield by cyanoethylation of benzimidazole with acrylonitrile in the presence of tetramethylammonium hydroxide. Reaction of I and RCH2CH2CN (R = piperidino, PhNEt, dipentylamino) with NH2OH gave the amidoximes or their dihydrochlorides.

ACCESSION NUMBER: 1997:458425 CAPLUS
DOCUMENT NUMBER: **127:108573**
TITLE: β-(Benzimidazol-1-yl)propionitrile and β-aminopropionic acid amidoximes
AUTHOR(S): Kayukova, L. A.; Dul'beeva, N. G.; Mirfaizova, R. Zh.
CORPORATE SOURCE: Inst. Khim. Nauk im. Bekturova, MN-AN RK, Almaty, Kazakhstan
SOURCE: Izvestiya Ministerstva Nauki--Akademii Nauk Respubliki Kazakhstan, Seriya Khimicheskaya (1996), (2), 55-60
CODEN: IMKKFL
PUBLISHER: Gylym
DOCUMENT TYPE: Journal
LANGUAGE: Russian
OTHER SOURCE(S): CASREACT 127:108573

L4 ANSWER 33 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Complexes between cobalt(III) and eight different 1,4,7,10-tetraazacyclododecane (cyclen) as well as two tris(3-aminopropyl)amine (trpn) derivs. are reported with varying nos. and structures of peralkylammonium groups in side chains of the ligands. The presence of addnl. pos. charges has small effects on hydrolysis rates of nitrophenyl- and bis(nitrophenyl)phosphate esters but leads to substantially enhanced cleavage of plasmid DNA. Increasing the number of the charged side groups and/or their distance to the metal ion center provides for better binding to the DNA groove, as shown also by affinity measurements with calf-thymus DNA. In line with this, saturation kinetics of plasmid DNA cleavage yield a corresponding increase of efficiency in Michaelis-Menten-type KM values, with rather constant kcat parameters. A binuclear cobalt complex with two cyclen centers separated by a -(CH2)6-N+(CH3)2-(CH2)6-N+(CH3)2-(CH2)6- spacer shows, with only 5+10-5 M catalyst concentration, the largest known rate enhancement factor of >107 (corresponding to >1011 at 1 M) against DNA; incubation with 0.05 mM at 37° for only 2 h leads to almost complete cleavage without appearance of products typical for redox cleavage. These results are in contrast to expts. with corresponding copper(II) complexes with added hydrogen peroxide, which has no effect with corresponding Co, Zn, Cd, or Ni complexes.

ACCESSION NUMBER: 1997:347191 CAPLUS
DOCUMENT NUMBER: **127:46819**
TITLE: Cobalt(III) Polyamine Complexes as Catalysts for the Hydrolysis of Phosphate Esters and of DNA. A Measurable 10 Million-Fold Rate Increase
AUTHOR(S): Hettich, Ronald; Schneider, Hans-Joerg
CORPORATE SOURCE: FR Organische Chemie der Universitaet des Saarlandes, Saarbruecken, D 66041, Germany
SOURCE: Journal of the American Chemical Society (1997), 119(24), 5638-5647
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 127:46819
REFERENCE COUNT: 64 THERE ARE 64 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 34 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB A simple and efficient total synthesis of triamines with selectively protected primary amino groups, i.e [Me3COCONH(CH2)nNHCH2(CH2)2NHCO2CH2Ph, n = 3-5], and triamines with the secondary amino function and in one of the primary amino functions protected, i.e. [EtOCONH(CH2)nN(CO2CMe3)(CH2)3 NH2, n = 3-5], was described.

ACCESSION NUMBER: 1996:514051 CAPLUS
DOCUMENT NUMBER: 125:248205
TITLE: Synthesis of carbamate-protected spermidine homologs through alkane-α,ω-diamines
AUTHOR(S): Araujo, M. Joao S. M. P.; Ragnarsson, Ulf; Trigo, M. Joaquina S. A. Amaral; Almeida, M. Lurdes S.
CORPORATE SOURCE: Cent. Invest. Quim. Univ. Porto, Fac. Cienc. Porto, Oporto, 4150, Port.
SOURCE: Journal of Chemical Research, Synopses (1996), (8), 366-367
CODEN: JRPSDC; ISSN: 0308-2342
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 125:248205

L4 ANSWER 35 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB RNR1(CH2)3NH2 [R, R1 = H, C1-22 (hydroxy)alkyl] are prepared by catalytic hydrogenation of RNR1(CH2)2CN in the presence of RNHR1. Autoclaving a mixture of 3-[N-(2-hydroxyethyl)-N-methylamino]propionitrile, MeNHCH2CH2OH, and Raney Ni at 60° and 30 kg/cm2-gage for 3 h to give 89% N-(2-hydroxyethyl)-N-methyl-1,3-propanediamine.

ACCESSION NUMBER: 1995:780699 CAPLUS
DOCUMENT NUMBER: 123:339135
TITLE: Preparation of N-(3-aminopropyl)amines
INVENTOR(S): Kaita, Jun; Ootawa, Yasunori; Kato, Tooru; Sotodani, Koshiro
PATENT ASSIGNEE(S): Kao Corp, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 07157453	A2	19950620	JP 1993-302605	19931202
JP 3224922	B2	20011105		
PRIORITY APPLN. INFO.:			JP 1993-302605	19931202

OTHER SOURCE(S): CASREACT 123:339135; MARPAT 123:339135

L4 ANSWER 36 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB A novel method derived from Kaden's modification (1983) of the Richman and Atkins's (1972) cyclization using tosylated synthons allows the unequivocal synthesis of 1- and 8-monofunctionalized 1,4,8,12-tetraazacyclopentadecane. Both syntheses are described.

ACCESSION NUMBER: 1995:353522 CAPLUS
DOCUMENT NUMBER: 122:187556
TITLE: First unequivocal synthesis of 1- or 8-N-monosubstituted 1,4,8,12-tetraazacyclopentadecane
AUTHOR(S): Granier, Colin; Guilard, Roger
CORPORATE SOURCE: Lab. Ing. Mol. Sep. Appl. Gaz, Fac. Sci. "Gabriel", Dijon, 21100, Fr.
SOURCE: Tetrahedron (1995), 51(4), 1197-208
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 122:187556

L4 ANSWER 37 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB HOCnH2nNR(CH2)3NH2 [I; R = H, C1-6 (hydroxy)alkyl; n = 2-9], useful as materials for surfactants, softeners, dyes, acidic gas removers, polymers, etc. (no data), are prepared by reduction of HOCnH2nNR(CH2)2CN (II; R, n = same as I) by H in presence of Raney Ni, NH3, and 0-50 weight% (based on II) C1-5 alcs. II (R = Me, n = 2) was treated with Raney Ni and NH3 under 20 kg/cm2-G H at 55-65° for 5 h to give 92% I (R = Me, n = 2).

ACCESSION NUMBER: 1994:163451 CAPLUS
DOCUMENT NUMBER: 120:163451
TITLE: Preparation of N-(3-aminopropyl)amines from amino nitriles
INVENTOR(S): Tatezawa, Osamu; Kitayama, Hiroaki; Kaita, Jun; Kato, Tooru; Sotodani, Koshiro
PATENT ASSIGNEE(S): Kao Corp, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 3 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05246959	A2	19930924	JP 1992-46779	19920304
JP 2951790	B2	19990920		
PRIORITY APPLN. INFO.:			JP 1992-46779	19920304

OTHER SOURCE(S): CASREACT 120:163451; MARPAT 120:163451

L4 ANSWER 38 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Title compds. were prepared by Michael addition of polyamines with acrylonitrile followed by reduction The reduction of polynitriles is achieved by the use of Dibal in high yield. Thus, N(CH2CH2NH2)3 was refluxed 24h in CH2:CHCN containing HOAC to give 81% N[CH2CH2N(CH2CH2CN)2]3 which was refluxed 24h with Dibal in THF/hexane to give 90% N[CH2CH2N(CH2CH2NH2)2]3 (I). I was further condensed with acrylonitrile. Condensation of I with 2-(HO)C6H4CHO gave a dendrimeric hexaimine which was used to form a tricobalt complex.

ACCESSION NUMBER: 1994:163403 CAPLUS
DOCUMENT NUMBER: 120:163403
TITLE: Preparation of dendrimeric polyamines
AUTHOR(S): Moors, Rolf; Voegtle, Fritz
CORPORATE SOURCE: Inst. Org. Chem. Biochem., Univ. Bonn, Bonn, D-53121, Germany
SOURCE: Chemische Berichte (1993), 126(9), 2133-5
CODEN: CHBEAM; ISSN: 0009-2940
DOCUMENT TYPE: Journal
LANGUAGE: German
OTHER SOURCE(S): CASREACT 120:163403

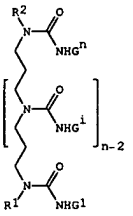
L4 ANSWER 39 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB New routes to open-chain polyamines have been developed using aliphatic amino azides as common precursors for the construction of the carbon-nitrogen framework. These α,ω-diaminoalkane synthetic equivalent were combined with ω-haloboranes to extend the polyamine chain from the azido moiety. An extension from the free amino group can also be achieved via a Michael type addition with acrylonitrile or a reductive amination with a γ-azido ketone. Further transformations led to a large variety of regioselectively C- and/or N-substituted polyamines.

ACCESSION NUMBER: 1993:516735 CAPLUS
DOCUMENT NUMBER: 119:116735
TITLE: Aliphatic amino azides as key building blocks for efficient polyamine syntheses
AUTHOR(S): Carboni, Bertrand; Benalil, Aziza; Vaultier, Michel
CORPORATE SOURCE: Univ. Rennes I, Campus de Beaulieu, Rennes, 35042, Fr.
SOURCE: Journal of Organic Chemistry (1993), 58(14), 3736-41
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 119:116735

L4 ANSWER 40 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

GI



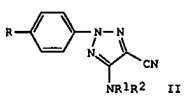
I

AB Synthetic and spectroscopic studies of di- and triurea derivs. I [n = 2, 3; R1, R2 = Ph, Et, CH2CH2CN; G1, G2, G3 = Ph, CH(CH2Ph)CO2Me, etc.] of 1,3-diaminopropane and 3,3'-iminobispropylamine are reported. These compds. are prepared efficiently by an iterative procedure involving three steps: (1) conjugate addition of a primary amine to acrylonitrile; (2) reaction of the resulting secondary amino group with an isocyanate; and (3) reduction of the nitrile group to generate a primary amine. Intramol. hydrogen bonding and substituents R1 and R2 provide extensive conformational control in ureas I. Hydrogen bonding results in large downfield shifts (0.58-2.43 ppm) of the NH resonances in the 1H NMR spectra of I. The IR spectra of I exhibit free and hydrogen bonded N-H stretches (3426-3463 cm-1 and 3284-3306 cm-1, resp.). Groups R1 and R2 control the orientation of the carbonyl groups. When R1 is Ph, the adjacent carbonyl group aligns in a trans orientation about the adjoining C-N bond. Intramol. hydrogen bonding aligns the other carbonyl groups in the same direction. 1H NMR and IR spectroscopy indicate that ca. 25-95% of I is intramolecularly hydrogen bonded in dilute chloroform solution

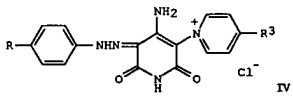
ACCESSION NUMBER: 1992:447621 CAPLUS
DOCUMENT NUMBER: 117:47621
TITLE: Molecular scaffolds. I. Intramolecular hydrogen bonding in a family of di- and triureas
AUTHOR(S): Nowick, James S.; Powell, Noel A.; Martinez, Eduardo J.; Smith, Eric M.; Noronha, Glenn
CORPORATE SOURCE: Dep. Chem., Univ. California, Irvine, CA, 92717, USA
SOURCE: Journal of Organic Chemistry (1992), 57(14), 3763-5
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 117:47621

L4 ANSWER 41 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

GI



II



IV

AB 4-RC6H4NHN:C(CN)C(:NH)NR1R2 (I, R = H, OMe, NO2, CO2Et, NR1R2 = morpholino; R = H, NR1R2 = NMe2, piperidino) were synthesized by reaction of 4-RC6H4NHN:C(CN)2 with secondary amines and used for subsequent cyclization reactions. Thus, I undergo cyclooxidn. by treatment with CuSO4/pyridine to form the 5-dialkylamino-2-aryl-1,2,3-triazolo-4-carbonitriles II. II (R = H, NR1R2 = morpholino) and N2H4-DMF gave the 4-(1,3,4-triazolyl-5)-1,2,3-triazole. The chloroacetylation of I is accompanied by hydrolysis of the amino group to yield 4-RC6H4NHN:C(CN)CONHCOCH2Cl (III). The quaternization of III with pyridines is followed by the Thorpe cyclization to form the 4-amino-5-arylazo-6-hydroxy-3-pyridiniopyrid-2-one chlorides IV (R3 = H, Me), useful as cationic dyes. The reaction of I with Cl3CCN yields 5-arylazo-4-imino-2-trichlormethyl-1,4-dihydropyrimidines which can be converted into the 2-hydrazinopyrimidine derivs.

ACCESSION NUMBER: 1991:429213 CAPLUS
DOCUMENT NUMBER: 115:29213
TITLE: Synthesis and reactions of 2-arylhydrazono-2-cyano-N,N-dialkylacetamidines
AUTHOR(S): Schaefer, H.; Gewald, K.; Bellmann, P.; Gruner, M.
CORPORATE SOURCE: Sekt. Chem., Tech. Univ., Dresden, 8027, Germany
SOURCE: Monatshefte fuer Chemie (1991), 122(3), 195-207
CODEN: MOCMB7; ISSN: 0026-9247
DOCUMENT TYPE: Journal
LANGUAGE: German
OTHER SOURCE(S): CASREACT 115:29213

L4 ANSWER 42 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB R1CH2CONHWNH2 (I) [R1 = ArO, aryl, heterocyclyl, etc.; Ar = (substituted) aryl, heterocyclyl, etc.; W = (CH2)n, (CH2)mNY(CH2)a; n, m, a = 2-6; Y = H, alkyl, (substituted) aryl, etc.; Z = H, C(:NH)NH2, CO2R2, etc.; R2 = alkyl] were prepared Reaction of N-(tert-butoxycarbonyl)-1,4-diaminobutane with 3- trifluoromethylphenoxyacetyl chloride, followed by deprotection in EtOH containing HCl, gave I.HCl [R1 = 3-trifluoromethylphenoxy; W = (CH2)4; Z = H]. N-[6-(sec-Butyl)-2,4-dichlorophenoxy]acetyldiamino-1,5-pentane at 500 ppm gave 70% control of Botrytis cinerea.

ACCESSION NUMBER: 1991:121758 CAPLUS
DOCUMENT NUMBER: **114:121758**
TITLE: Preparation of aryl- and aryloxyacetyldiaminoalkanes and analogs as agrochemical fungicides
INVENTOR(S): Brayer, Jean Louis; Taliani, Laurent; Tessier, Jean
PATENT ASSIGNEE(S): Roussel-UCLAF, Fr.
SOURCE: Eur. Pat. Appl., 41 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

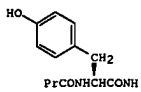
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 376819	A1	19900704	EP 1989-403614	19891222
R: BE, CH, DE, ES, FR, GB, IT, LI, LU, NL				
FR 2642422	A1	19900803	FR 1988-16994	19881222
FR 2642422	B1	19940713		
US 5064861	A	19911112	US 1989-454685	19891221
JP 02225450	A2	19900907	JP 1989-331482	19891222

PRIORITY APPLN. INFO.: FR 1988-16994 A 19881222

OTHER SOURCE(S): CASREACT 114:121758; MARPAT 114:121758

L4 ANSWER 43 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

GI



PrCONHCHCONH(CH2)4NH(CH2)3NH(CH2)3NH2 I

AB The title polyamine toxin (I) present in the venom sac of the solitary digger wasp Philanthus triangulum was isolated, characterized, and synthesized. PhTX-433, having a butyltyrosylpolyamine structure, is a potent noncompetitive inhibitor of the quisqualate subtype glutamate receptor as assayed by the twitch contraction of locus leg muscles. It is also an allosteric inhibitor of acetylcholine receptors of vertebrates. About 60 analogs of I were synthesized to clarify structure/activity relations and to select candidates suitable for isolating the glutamate receptor by photoaffinity label and/or affinity label.

ACCESSION NUMBER: 1990:606432 CAPLUS
DOCUMENT NUMBER: **113:206432**
TITLE: Philanthotoxin-433 (PhTX-433), a non-competitive glutamate receptor inhibitor
AUTHOR(S): Nakanishi, Koji; Goodnow, R.; Konno, K.; Niwa, M.; Bukownik, Rudolph; Kallimopoulos, Thomas A.; Usherwood, Peter; Eldefrawi, Amira T.; Eldefrawi, Mohyee E.
CORPORATE SOURCE: Dep. Chem., Columbia Univ., New York, NY, 10027, USA
SOURCE: Pure and Applied Chemistry (1990), 62(7), 1223-30
CODEN: PACHAS; ISSN: 0033-4545
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 113:206432

L4 ANSWER 44 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB The synthesis of the non-competitive glutamate receptor inhibitor, philanthotoxin 433, PrCO-Tyr-NHCH2(CH2CH2NH)3H, (PhTX-433) is described. Two synthetic routes are described for the preparation of a variety of structural analogs.

ACCESSION NUMBER: 1990:591878 CAPLUS
DOCUMENT NUMBER: **113:191878**
TITLE: Synthesis of glutamate receptor antagonist philanthotoxin-433 (PhTX-433) and its analogs
AUTHOR(S): Goodnow, R., Jr.; Konno, K.; Niwa, M.; Kallimopoulos, T.; Bukownik, R.; Lenares, Deborah; Nakanishi, K.
CORPORATE SOURCE: Dep. Chem., Columbia Univ., New York, NY, 10027, USA
SOURCE: Tetrahedron (1990), 46(9), 3267-86
CODEN: TETRAB; ISSN: 0040-4020
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 113:191878

L4 ANSWER 45 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN

AB Eleven lipophilic derivs. of the title biogenic amines and 28 structurally related triamines and tetramines have been synthesized. Twenty-three of them inhibited the platelet aggregation induced by collagen at an ED50 of 8-30 μmol/L. Five compds. prolonged the one-stage thromboplastin time (Quick) by 7s or more at 100 μmol/L. The antiplatelet and anticoagulant effects do not run parallel. The relationship between the effects observed and the chemical structure of the oligoamines has been elucidated.

ACCESSION NUMBER: 1990:497320 CAPLUS
DOCUMENT NUMBER: **113:97320**
TITLE: Platelet aggregation inhibiting and anticoagulant effects of oligoamines. XII. Alkyl and aralkyl derivatives of putrescine, spermidine, and spermine
AUTHOR(S): Rehse, Klaus; Puchert, Eckhard; Leissring, Susanne
CORPORATE SOURCE: Inst. Pharm., Freie Univ. Berlin, Berlin, 1000/33, Germany
SOURCE: Archiv der Pharmazie (Weinheim, Germany) (1990), 323(5), 287-94
CODEN: ARPMAS; ISSN: 0365-6233
DOCUMENT TYPE: Journal
LANGUAGE: German
OTHER SOURCE(S): CASREACT 113:97320

L4 ANSWER 46 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
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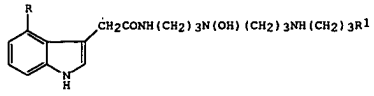
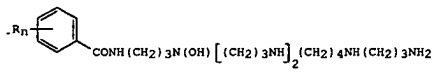


I, R=H, R^1=$NH(CH_2)_4NH(CH_2)_3NH_2$
II, R=OH, R^1=$NH(CH_2)_4NH(CH_2)_3NH_2$
V, R=OH, R^1=$CH_2NH(CH_2)_3NH_2$



III, R_n=4-HO
IV, R_n=2,5-$(HO)_2$

AB Novel hydroxylamine-containing polyamines were isolated from the venom of A.
aperta, a funnel-web spider found throughout the western United States. 1H and 13C NMR in concert with UV spectroscopy provided important structural inputs, particularly for determining the aromatic chromophores.
Fast-atom bombardment (FAB) mass spectrometry, however, provided the key data for establishing the polyamine functionality of Agel 489 (I), 505 (II), 452 (III), and 448 (IV). These polyamine structures were confirmed by total synthesis. The recurring propylamine portions of the polyamine chains were assembled by amine cyanoethylation followed by nitrile reduction
Hydroxylamine incorporation was best achieved via a 2-step sequence involving amine oxidation with 2-(phenylsulfonyl)-3-phenyloxazidirine followed by sodium cyanoborohydride reduction of any nitrone byproducts. TFA
and(or) dioxane/HCl treatment of the tert-butoxycarbonyl (BOC) and methyloxymethoxy (MOM) protected intermediates provided the natural Agelenopsis hydroxylamine products as their acid salts. In the HCl-mediated removal of the protecting groups to generate II and V, intermediate 1-carboxyl-3-indoleacetamides were isolated.
ACCESSION NUMBER: 1990:495011 CAPLUS
DOCUMENT NUMBER: **113:95011**
TITLE: Isolation, structure elucidation, and synthesis of novel hydroxylamine-containing polyamines from the venom of the Agelenopsis aperta spider
AUTHOR(S): Jasys, V. John; Kelbaugh, Paul R.; Nason, Deane M.; Phillips, Douglas; Rosnack, Kenneth J.; Saccomano, Nicholas A.; Stroh, Justin G.; Volkmann, Robert A.
CORPORATE SOURCE: Pfizer Inc., Groton, CT, 06340, USA
SOURCE: Journal of the American Chemical Society (1990), 112(18), 6696-704
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 113:95011

L4 ANSWER 47 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB A series of tetraamines derived from 1,8-diaminooctane was prepared and tested as antitumor agents. The reaction of 1,8-diaminooctane with acrylonitrile gave N,N'-bis(cyanoethyl)-1,8-diaminooctane, which was reduced to tetraamine. Alkylation of the terminal nitrogen atoms of the tetra-Boc derivative of this compound by Me or Et halide followed by removal of
the Boc groups gave the bis(alkyl)polyamines, resp. These compds. exhibit
promising antitumor activity in the mouse L1210 leukemia model. Coadministration of a polyamine oxidase inhibitor potentiated the antitumor activity.
ACCESSION NUMBER: 1990:197554 CAPLUS
DOCUMENT NUMBER: **112:197554**
TITLE: Polyamine analogs with antitumor activity
AUTHOR(S): Edwards, Michael L.; Prakash, N. J.; Stemerick, D. M.; Sunkara, S. P.; Bitonti, A. J.; Davis, G. F.; Dumont, J. A.; Bey, P.
CORPORATE SOURCE: Merrell Dow Res. Inst., Cincinnati, OH, 45215, USA
SOURCE: Journal of Medicinal Chemistry (1990), 33(5), 1369-75
CODEN: JMCMAR; ISSN: 0022-2623
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 112:197554

L4 ANSWER 48 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB The title compound (I), useful as a chain extender for polyurethanes, is prepared with high yield, selectivity, and purity by adding acrylonitrile (II) to HCONH2 in the presence of 4-aminopyridine derivs. and hydrogenating the resulting dinitrile. Refluxing II 212, HCONH2 180, 4-(dimethylamino)pyridine 14.7, and MeCN 212 g for 21 h gave 170 g HCON(CH2CH2CN)2 (III) with purity 99% and selectivity 95%. Hydrogenating 255 g III in MeOH-NH3 over 40 g Raney Fe-Ni (15:85) at 30°/30-50 bar gave 262 g I (.apprx.50:50 mixture of N1 and N2 isomers).
ACCESSION NUMBER: 1990:119575 CAPLUS
DOCUMENT NUMBER: **112:119575**
TITLE: Bis(trimethylene)triamine monoformamide manufacture
INVENTOR(S): Scholl, Hans Joachim; Reiff, Helmut
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.
SOURCE: Ger. Offen., 6 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3811342	A1	19891012	DE 1988-3811342	19880402
CA 1309728	A1	19921103	CA 1989-593838	19890315
EP 336184	A1	19891011	EP 1989-104900	19890318
EP 336184	B1	19930113		
R: CH, DE, FR, GB, IT, LI, NL				
US 4923952	A	19900508	US 1989-329472	19890328
JP 02006444	A2	19900110	JP 1989-78757	19890331
JP 2583450	B2	19970219		
PRIORITY APPLN. INFO.:			DE 1988-3811342 A	19880402

OTHER SOURCE(S): CASREACT 112:119575; MARPAT 112:119575

L4 ANSWER 49 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB Me2N(CH2)3NH2 (I) is prepared by hydrogenation of Me2NCH2CH2CN (II) in the presence of an alkaline earth oxide which suppresses formation of H2N(CH2)3NH2 (III). Thus, II, Raney Co, and NH3 (liquid) were charged to an autoclave and the whole maintained at 160° and 150 bar H to give I containing <50 ppm III.

ACCESSION NUMBER: 1989:614118 CAPLUS
DOCUMENT NUMBER: 111:214118
TITLE: Process for the preparation of N,N-dimethyldiaminopropane
INVENTOR(S): Kiel, Wolfgang; Bauer, Wolfgang
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.
SOURCE: Eur. Pat. Appl., 3 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 316761	A2	19890524	EP 1988-118720	19881110
EP 316761	A3	19900704		
R: CH, DE, FR, GB, LI				
DE 3739260	A1	19890601	DE 1987-3739260	19871118
PRIORITY APPLN. INFO.:			DE 1987-3739260 A	19871118

OTHER SOURCE(S): CASREACT 111:214118

L4 ANSWER 50 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB Exptl. design data for the synthesis of N,N-dimethyl-1,3-propanediamine (I) by catalytic hydrogenation of β-dimethylaminopropionitrile (II) showed that optimum conversion of II at maximum I yield I can be obtained at H-II mol ratio ≥10 and 106-130°. In an exptl. verification of the process at H-II mol ratio 10 and 110°, II conversion was 95% and I yield was 91%.

ACCESSION NUMBER: 1989:576694 CAPLUS
DOCUMENT NUMBER: 111:176694
TITLE: Search for optimal conditions for synthesis of N,N-dimethyl-1,3-propanediamine by the experimental design method
AUTHOR(S): Popovich, O. T.; Pavlenko, N. V.; Golodets, G. I.
CORPORATE SOURCE: Kiev. Politekh. Inst., Kiev, USSR
SOURCE: Khimicheskaya Tekhnologiya (Kiev) (1989), (4), 58-61
CODEN: KHMTA6; ISSN: 0368-556X
DOCUMENT TYPE: Journal
LANGUAGE: Russian
OTHER SOURCE(S): CASREACT 111:176694

L4 ANSWER 51 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB Addition of 0.03-0.1 weight% of diethylaminopropiothioamide to an absorbent for removing acid impurities from hydrocarbon gas increases the absorption capacity during prolonged use and decreases the H absorption by metal. The absorbent also contains 5-50 weight% of an aqueous solution of alkanolamine and 0.005-1.0 weight% of tertiary aminonitrile, the balance being water.

ACCESSION NUMBER: 1989:157549 CAPLUS
DOCUMENT NUMBER: 110:157549
TITLE: Absorbent for purifying hydrocarbon gas
INVENTOR(S): Mitina, A. P.; Legezin, N. E.; Gollandskikh, N. I.; Frolova, L. V.; Brusnikina, V. M.; Aminov, M. Kh.; Galeeva, R. G.; Levanov, V. V.; Sakhapov, Ya. M.
PATENT ASSIGNEE(S): All-Union Scientific-Research Institute of Natural Gas, USSR; Institute of Chemical Physics, Academy of Sciences, U.S.S.R.
SOURCE: U.S.S.R. From: Otkrytiya, Izobret. 1988, (43), 39.
CODEN: URXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Russian
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
SU 1438831	A1	19881123	SU 1987-4232010	19870420
PRIORITY APPLN. INFO.:			SU 1987-4232010	19870420

OTHER SOURCE(S): CASREACT 110:157549

L4 ANSWER 52 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB The efficiencies of three rigidly held cis-aquohydroxotetraazacobalt(III) complexes [(cyclen)Co(OH)(OH2)]2+ (cyclen = 1,4,7,10-tetraazacyclododecane), [(tren)Co(OH)(OH2)]2+ [tren = N(CH2CH2NH2)3], and [(trpn)Co(OH)(OH2)]2+ [trpn = N(CH2CH2CH2NH2)3] in promoting the hydrolysis of bis(p-nitrophenyl) phosphate (BNPP) have been compared. In neutral water at 50°, the rate constant for hydrolysis of the phosphate diester bond in [(cyclen)Co(OH)(BNPP)]+, [(tren)Co(OH)(BNPP)]+, [(trpn)Co(OH)(BNPP)]+ are 4.6 + 10-1, 8.1 + 10-3, and 2.5 s-1, resp. [(Trpn)Co(OH)(BNPP)]+ is hydrolyzed at about the same rate as BNPP bound to a real enzyme from Enterobacter aerogenes and about 1010 times more rapidly than free BNPP. The dramatic increase in the activity of the Co(III) complex with change in the tetraamine ligand structure can be explained in terms of a detailed mechanism of the reaction.

ACCESSION NUMBER: 1989:39085 CAPLUS
DOCUMENT NUMBER: 110:39085
TITLE: Cobalt(III) complex-promoted hydrolysis of phosphate diesters: comparison in reactivity of rigid cis-diaquo(tetraaza)cobalt(III) complexes
AUTHOR(S): Chin, Jik; Banaszczyk, Mariusz; Jubian, Vrej; Zou, Xiang
CORPORATE SOURCE: Dep. Chem., McGill Univ., Montreal, QC, H3A 2K6, Can.
SOURCE: Journal of the American Chemical Society (1989), 111(1), 186-90
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 110:39085

L4 ANSWER 53 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
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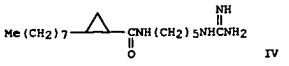

IV

AB Title compds. R1CONR2XNHR3 [I; R1 = C1-20 alkyl, alkenyl, aryl, aralkyl, cycloalkyl, alkylcycloalkyl, cycloalkylalkyl, cycloalkenyl, alkylcycloalkenyl, cycloalkenylalkyl; R2 = H, C1-10 alkyl, C≤10 amide-substituted alkyl; R3 = C(:NR4)NR5R6, 4,6-dimethyl-2-pyrimidinyl, CONH2; R4-R6 = H, C1-8 alkyl; X = C2-12 hydrocarbondiyl, especially (CH2)n where n = 2-12; R2 and X may form alkylene chain] are prepared as bactericides (no data). Condensation of H2N(CH2)5NH2 with MeSC(:NH)NH2.H2SO4 in H2O gave H2N(CH2)5NHC(:NH)NH2 (II). Cyclopropanation of 1-decene by N2CHCO2Et in the presence of CuSO4, followed by saponification of the resultant ester and treatment with SOCl2, gave 2-(n-octyl)cyclopropanecarbonyl chloride (III). Acylation of II by III in pyridine gave carbamimidoyl(octylcyclopropanecarbonyl)pentamethylenediamine IV.

ACCESSION NUMBER: 1988:610572 CAPLUS
DOCUMENT NUMBER: **109:210572**
TITLE: Acyl(carbamimidoyl)alkanediamines useful as bactericides
INVENTOR(S): Rinehart, Kenneth L., Jr.; Carter, Guy T.; Cheng, Michael T.
PATENT ASSIGNEE(S): University of Illinois Foundation, USA
SOURCE: U.S., 5 pp. Cont. of U.S. Ser. No. 50,139, abandoned. CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4762949	A	19880809	US 1983-460287	19830124
PRIORITY APPLN. INFO.:			US 1979-50139 A1	19790620

OTHER SOURCE(S): CASREACT 109:210572; MARPAT 109:210572

L4 ANSWER 54 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB Review with 301 refs. 3-Dimethylaminopropylamine (I) is prepared by the addition of Me2NH to CH2:CHCN and hydrogenation of Me2NCH2CH2CN. As a reasonably-priced bifunctional compound with differing reactivity in the primary and tertiary amino groups I is suitable for the manufacture of secondary products with interesting properties for industrial applications, e.g. hardeners and bonding agents, ion exchangers, additives for flocculants, cosmetics and fuel, dyes and pesticides.

ACCESSION NUMBER: 1988:422504 CAPLUS
DOCUMENT NUMBER: **109:22504**
TITLE: 3-(Dimethylamino)propylamine as an actual key reactant
AUTHOR(S): Lappe, Peter; Springer, Helmut; Weber, Juergen
CORPORATE SOURCE: Ruhrchem. A.-G., Oberhausen, D-4200/11, Fed. Rep. Ger.
SOURCE: Chemiker-Zeitung (1987), 111(4), 117-25 CODEN: CMKZAT; ISSN: 0009-2894
DOCUMENT TYPE: Journal; General Review
LANGUAGE: German
OTHER SOURCE(S): CASREACT 109:22504

L4 ANSWER 55 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB Spermine derivs. RR1N(CH2)3NR2(CH2)4NR2(CH2)3NRR1 [I, R = Me, Et, Pr, (CH2)3NHEt, R1 = R2 = H; R = R1 = Et, R2 = H; R = H, Et, R1 = H, R2 = Et), EtNH(CH2)12NHEt (II), and 1,4-bis(3-ethylaminopropyl)piperazine were prepared I (R = Me, Et, Pr, R1 = R2 = H) inhibit the growth of L1210 cells, in culture with ED <1 μM at 96 h. Furthermore, I (R = Et, R1 = R2 = H) is similarly active against Daudi and HL-60 cells in culture. A structure-activity relationship exists between the position at which spermine is alkylated and its antiproliferative properties. At 10μM I (R = Et, R1 = R2 = H) was cytostatic against L1210 cells with >90% cell viability by trypan blue exclusion, even after a 144-h exposure. When L1210 cells were treated with 10 μM I (R = Et, R1 = R2 = H) over a 144-h period, their size and mitochondrial DNA content were gradually but substantially diminished. However, flow cytometric measurements of the nuclear DNA content of these treated cells at 96 h indicated only slightly reduced S and G2 populations and significant changes only after 144 h. A cloning assay performed on the cells after 96 h of exposure to 10 μM I (R = Et, R1 = R2 = H) indicated that the cells were not growing. When mice, inoculated with L1210 leukemia, were treated with I (R = Et, R1 = R2 = H) their life span was increased >200% relative to controls. Moreover, many long-term survivors were apparently tumor-free at the end of 60 days.

ACCESSION NUMBER: 1988:221482 CAPLUS
DOCUMENT NUMBER: **108:221482**
TITLE: Synthetic polyamine analogs as antineoplastics
AUTHOR(S): Bergeron, Raymond J.; Neims, Allen H.; McManis, James S.; Hawthorne, Thomas R.; Vinson, John R. T.; Bortell, Rita; Ingeno, Michael J.
CORPORATE SOURCE: Dep. Med. Chem., Univ. Florida, Gainesville, FL, 32610, USA
SOURCE: Journal of Medicinal Chemistry (1988), 31(6), 1183-90 CODEN: JMCMAR; ISSN: 0022-2623
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 108:221482

L4 ANSWER 56 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
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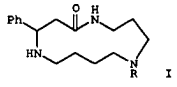

I

AB (S)-3-Amino-3-phenylpropionic acid was converted to dihydrocelacinnine (I, R = PhCH2CH2CO) and celabenzine (I, R = PhCO).

ACCESSION NUMBER: 1987:120128 CAPLUS
DOCUMENT NUMBER: **106:120128**
TITLE: Total synthesis of (-)-dihydrocelacinnine and (±)-celabenzine
AUTHOR(S): Iida, Hideo; Fukuhara, Kiyoshi; Machiba, Mitsuo; Kikuchi, Toyohiko
CORPORATE SOURCE: Tokyo Coll. Pharm., Hachioji, 192-03, Japan
SOURCE: Tetrahedron Letters (1986), 27(2), 207-10 CODEN: TELEAY; ISSN: 0040-4039
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 106:120128

L4 ANSWER 57 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
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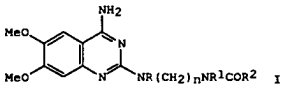

AB N2-[(Acylamino)alkyl]-6,7-dimethoxy-2,4-quinazolinediamines I (e.g., R, R1, R2, n = Me, Me, Ph, 2; Me, H, tetrahydro-2-furyl, 3) were synthesized as potential α1-adrenoceptor antagonists. In rats at 10 mg/kg po, some I (n = 3) showed good antihypertensive activity, whereas I (n = 2) did not.

ACCESSION NUMBER: 1986:148830 CAPLUS
DOCUMENT NUMBER: **104:148830**
TITLE: Synthesis and antihypertensive activity of a series of 4-amino-6,7-dimethoxyquinazoline derivatives
AUTHOR(S): Manoury, Philippe M.; Binet, Jean L.; Dumas, Andre P.; Lefevre-Borg, Francoise; Cavero, Icilio
CORPORATE SOURCE: Chem. Dep., Lab. Etud. Rech. Synthelabo, Bagneux, 92220, Fr.
SOURCE: Journal of Medicinal Chemistry (1986), 29(1), 19-25
CODEN: JMCMAR; ISSN: 0022-2623
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 104:148830

L4 ANSWER 58 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
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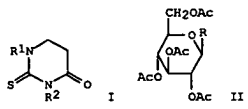

AB Reaction of aminopropanenitriles R1NHCH2CH2CN (R1 = cyclohexyl, Bu, Et) with isothiocyanates R2NCS (R2 = Ph, Et, Bz) in C6H6 at room temperature gave 92-100% adducts R1N(CH2CH2CN)CSNHR2 (same R1 and R2), which on refluxing in Me2CO-H2O in the presence of HCl gave 64-96% dihydrothiouracils I (same R1; R2 = Ph, Et, H). Glucopyranosyl isothiocyanate (II; R = NCS), obtained from 2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl bromide and Pb(SCN)2, on refluxing with EtNH(CH2)2CN in ether gave 74% thiourea derivative [II; R = NHCSN[(CH2)2CN]Et], which on refluxing in Me2CO containing HCl gave 72% II [R = NHCSN[(CH2)2CONH2]Et]. The expected thiouracil derivative was not formed.

ACCESSION NUMBER: 1985:578225 CAPLUS
DOCUMENT NUMBER: **103:178225**
TITLE: Synthesis of 5,6-dihydro-2-thiouracils
AUTHOR(S): Yamamoto, Iwao; Fukui, Kenichi; Yamamoto, Sadao; Ohta, Kazuchika; Matsuzaki, Kei
CORPORATE SOURCE: Fac. Text. Sci. Technol., Shinshu Univ., Nagano, 386, Japan
SOURCE: Synthesis (1985), (6-7), 686-8
CODEN: SYNTBF; ISSN: 0039-7881
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 103:178225

L4 ANSWER 59 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB NC(CH2)nNR(CH2)mCN (R = H, CH2Ph, Me; m, n = 2,3), NCCH2CH2NH(CH2)4NHCH2CH2CN, and PhCH2NHCH2CH2CN were reduced by H in the presence of Raney Ni and NaOH to give H2N(CH2)n+1NR(CH2)m+1NH2. Debenzylation was avoided by the use of this catalyst.

ACCESSION NUMBER: 1985:95459 CAPLUS
DOCUMENT NUMBER: **102:95459**
TITLE: Amines and polyamines from nitriles
AUTHOR(S): Bergeron, Raymond J.; Garlich, Joseph R.
CORPORATE SOURCE: Dep. Med. Chem., Univ. Florida, Gainesville, FL, 32610, USA
SOURCE: Synthesis (1984), (9), 782-4
CODEN: SYNTBF; ISSN: 0039-7881
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 102:95459

L4 ANSWER 60 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB Selected homologs, analogs, and acylated derivs. of spermine and spermidine, together with several heterocyclic and aromatic compds. containing a novoldiamine side chain, were prepared and evaluated biol. Several compds. possessed activity against B-16 melanoma and human epidermoid carcinoma of the nasopharynx. Thus, HN(CH2CH2CN)2 was treated with palmitoyl chloride followed by catalytic reduction to give Me(CH2)14CON(CH2CH2NH2)2.

ACCESSION NUMBER: 1981:603242 CAPLUS
DOCUMENT NUMBER: **95:203242**
TITLE: Synthesis of new polyamine derivatives for cancer chemotherapeutic studies
AUTHOR(S): Weinstock, Louis T.; Rost, William J.; Cheng, C. C.
CORPORATE SOURCE: Midwest Res. Inst., Kansas City, MO, 64110, USA
SOURCE: Journal of Pharmaceutical Sciences (1981), 70(8), 956-9
CODEN: JPMSAE; ISSN: 0022-3549
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 95:203242

L4 ANSWER 61 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
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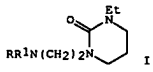

AB 1-Ethyl-hexahydropyrimidin-2-ones I (RR1N = morpholino, pyrrolidino, 4-methylpiperazino) were prepared and tested against Litomosoides carinii infection in cotton rats. None of them shows antifilarial activity at a dose of 30 mg/kg given for 6 days.

ACCESSION NUMBER: 1981:443023 CAPLUS
DOCUMENT NUMBER: **95:43023**
TITLE: Studies in potential filaricides. Part XII. A convenient synthesis of 1-ethyl-3-(2-substituted ethyl)hexahydropyrimidin-2-ones
AUTHOR(S): Dubey, Sushil Kumar; Sharma, Satyavan; Iyer, R. N.; Anand, Nitya
CORPORATE SOURCE: Div. Med. Chem., Cent. Drug Res. Inst., Lucknow, 226 001, India
SOURCE: Indian Journal of Chemistry, Section B: Organic Chemistry Including Medicinal Chemistry (1981), 20B(2), 170-1
CODEN: IJSBDB; ISSN: 0376-4699
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 95:43023

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AB Diacetylspermidine, AcNH(CH2)4NAc(CH2)3NH2 (I), was prepared by reducing AcNH(CH2)4NAc(CH2)2CN (II) with NaBH4, whereas H2N(CH2)4-β-Ala-OH was prepared by hydrolyzing II with HCl(g)-EtOH/1N HCl at room temperature and deacylating the resulting AcNH(CH2)4NAc(CH2)2CO2H with 6N HCl at 100°. H2N(CH2)4NH2 (1 mol) was monoacetylated with Ac2O (1 mol) and treated with CH2:CHCN to give AcNH(CH2)4NH(CH2)2CN, which was acylated with Ac2O to give II. I can be used in the synthesis of edeine antibiotics.

ACCESSION NUMBER: 1979:204452 CAPLUS
DOCUMENT NUMBER: **90:204452**
TITLE: Synthesis of N-(3-aminopropyl)-N,N'-diacetyl-1,4-diaminobutane and N-(4-aminobutyl)-3-aminopropionic acid
AUTHOR(S): Andruszkiewicz, Ryszard; Grzybowska, Jolanta; Wojciechowska, Hanna
CORPORATE SOURCE: Inst. Org. Food Chem. Technol., Polytech. Univ., Gdansk, Pol.
SOURCE: Polish Journal of Chemistry (1978), 52(11), 2251-4
CODEN: PJCHDQ; ISSN: 0137-5083
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 90:204452

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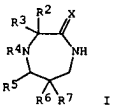

AB Diazepines I (R2, R3, R5-R7 = H, Me; R4 = Me, PhCH2, H; X = O) were obtained in 40-82% yields by reductive cyclization of R1O2CCR2R3NR4CHR5CR6R7CN (R1 = Me, Et). Reduction of I (X = O) by LiAlH4 gave 35-68% I (X = H2), which could be methylated, benzylated, or nitrosated.

ACCESSION NUMBER: 1978:546876 CAPLUS
DOCUMENT NUMBER: **89:146876**
TITLE: Synthesis of model substances for studies on conformation of heterocyclic heptanuclear rings. Part I. Synthesis of C-methyl derivatives of hexahydro-1,4-diazepine
AUTHOR(S): Majchrzak, Michal; Kotelko, Antoni; Guryn, Roman
CORPORATE SOURCE: Pharm. Fac., Sch. Med. Lodz, Lodz, Pol.
SOURCE: Polish Journal of Chemistry (1978), 52(5), 1023-8
CODEN: PJCHDQ; ISSN: 0137-5083
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 89:146876

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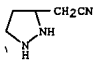

AB H2NNH2 reacts with H2C:CHCH:CHCN to give 3-(cyanomethyl)pyrazolidine (I), which was reduced to 1,3,5-pentanetriamine. I gave 1:1 and 1:2 open-chain and cyclic adducts. Treating 1- and 2-cyanobutadiene with amines gave from 1:1 to 4:3 adducts depending on the nature of the amine and the reaction conditions.

ACCESSION NUMBER: 1978:89574 CAPLUS
DOCUMENT NUMBER: **88:89574**
TITLE: Polyamines from cyanobutadienes
AUTHOR(S): Weigert, F. J.
CORPORATE SOURCE: Cent. Res. Dev. Dep., E. I. du Pont de Nemours and Co., Wilmington, DE, USA
SOURCE: Journal of Organic Chemistry (1978), 43(4), 622-6
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 88:89574

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GI For diagram(s), see printed CA Issue.
AB By esterification of N,N'-bis(ω-hydroxyalkyl)piperazines and N,N'-bis(ω-hydroxyalkyl)-N,N'-dialkylpolymethylenediamines with 3,4,5-(MeO)3C6H2CO2H (I) and related alkoxybenzoic and alkoxyphenylacetic acids were prepared 48 sym. bis esters. Several of these open chain bis compds. showed a noteworthy pharmacodynamic action, particularly dilation of the coronary vessels. Multiple synthetic variations within this class of compds. demonstrated that the action was closely allied with entirely specific structural elements and that therefore a high specificity existed in the relation between constitution and action. (Distns. of smaller amts. were carried out by bulb to bulb distns.; b.ps. reported which range over 10° or more refer to such distns. and are air bath temps.) The required acid chlorides were prepared in the usual way with SOCl2. 3,4,5-(MeO)3C6H2CH2CO2H was prepared from 3,4,5-(MeO)3C6H2COCl (Ia) by Arndt-Eistert reaction. 3,4,5-(MeO)2[3,4,5-(MeO)3C6H2CO2]C6H2COCl (II), m. 138-43°, was prepared from the corresponding acid (IIa) and SOCl2 in 79% yield. Acylations with II in the presence of C5H5N resulted also in preparation of the anhydride of IIa, m. 245-8°, also prepared from II and the Et3N salt of IIa. Methods A. A mixture of 0.05 mole acid chloride (with higher melting acid chlorides the reaction was preferably carried out in absolute C6H6) and 0.055 mole appropriate halo alc. heated gradually to 100° (HCl evolved at 50-60° and the mixture became homogeneous), the product heated 3 hrs. on a water bath and diluted with Et2O, the solution washed with aqueous NaHCO3, dried, and evaporated, and the residue recrystd. from Et2O-petr. ether gave over 70% the following 3,4,5-RR1R2C6H2CO2(CH2)nY (III) (R, R1, R2, n, Y, m.p., b.p./mm. given): MeO, MeO, MeO, 2, Cl, 73-4°, -; H, MeO, H, 3, Cl, -, 100-10°/0.1; MeO, MeO, H, 3, Cl, 52-3°, -; MeO, H, MeO, 3, Cl, -, 160-70°/0.2; Cl, MeO, H, 3, Cl, -, 130-40°/0.4; Cl, MeO, Cl, 3, Cl, -, 110-20°/0.1; MeO, MeO, MeO, 3, Cl, 57-9°, -; MeO, MeO, MeO, 3, Br, 63-6°, -; MeO, MeO, MeO, 3, I, 53-7°, -; EtO, EtO, EtO, 3, Cl, 57-60°, -; MeO, MeO, MeO, 4, Cl, -, 160-70°/0.4. Also prepared were the following 3,4,5-RR1R2C6H2CH2CO2(CH2)nY (IV) (same data): H, MeO, H, 2, Cl, -, 90-5°/0.1; MeO, MeO, MeO, 2, Cl, 37-40°, -; H, MeO, H, 3, Cl, -, 105-10°/0.2; MeO MeO, MeO, 3, Cl, 18-20°, 165-70°/0.1. Likewise prepared was 85% 3,4,5-(MeO)3C6H2CO2CH2C.tplbond.CCH2Cl, m. 84-5° (prepared from Ia and ClCH2C.tplbond.CCH2OH in C6H6 containing absolute C5H5N at room temperature). (1) III or IV (preferably the bromo compound) (0.1 mole) and 0.1 mole anhydrous piperazine or N,N'-dialkylethylenediamine in 100 ml. dry HCONMe2 (DMF) heated 24 hrs. on a water bath, DMF evaporated at 10 mm., the residue partitioned between EtOAc and H2O, the organic phase separated, washed with H2O, and extracted with 2N HCl, the extract saturated with K2CO3 and extracted with Et2O (or C6H6 or EtOAc, according to the solubility of the product), and the extract dried and evaporated gave 40-80% bis ester, which was contaminated with difficulty separable monoester; the piperazine derivs. were recrystd. from MeOH-Et2O, with addition of petr. ether when necessary, and the noncrystg.

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dialkylethylenediamine derivs. were converted into salts. (2) The reaction was carried out without solvent by heating the components 20-4 hrs. at 100°, the product dissolved in EtOAc-Et2O, and the soln. filtered and worked up as in 1. (3) A soln. of 0.05 mole diamine in 50 ml. abs. DMF treated with 2.9 g. NaH, the mixt. heated 15 min. on a water bath, 0.1 mole III or IV added, the mixt. heated 2 hrs. at 100° and filtered, and the filtrate worked up as in 1 gave better yields of purer bis compds. Methods B. (1) Ethylene oxide (9.7 g.) introduced into 50 ml. MeOH contg. 8.8 g. (MeNHCH2)2 (V) with ice cooling, when the exothermic reaction subsided the cooling bath removed, and the soln. kept 1 hr. at room temp., heated 15 min. on a water bath, and fractionated gave 9.85 g. (HOCH2CH2NMeCH2)2, b0.04 105-7°, n20D 1.4828; dipicrate m. 220°. Similarly was prepd. 65% HO(CH2)2NMe(CH2)3NMe(CH2)2OH, b0.1 100-10°, n20D 1.4773; dipicrate m. 152-6°. (2) A hot (70-80°) soln. of 11.0 g. Na in 82 ml. CH2:CHCH2OH treated with 19.5 g. V, the mixt. heated 100 hrs. at 100-5° (bath), cooled, dild. with 100 ml. H2O, and extd. with Et2O, and the ext. fractionated gave 6.0 g. HO(CH2)3NMeCH2CH2NHMe, b0.3-0.5 65-85° (dimethiodide m. 165-8°), and 17.1 g. [HO(CH2)3NMeCH2]2 (Va), b0.1 124-8°, n20D 1.4830 (dimethiodide m. 200-5°; dipicrate m. 148-9°). (3) Piperazine or dialkylpolymethylenediamine (0.1 mole) treated gradually with 0.25 mole CH2:CHCO2Me (VI), when the exothermic reaction subsided the mixt. heated 1 hr. on a water bath, treated with 0.5 mole VI, heated 1 hr. more, and evapd. in vacuo, and the residue recrystd. (piperazine derivs.) from Et2O-petr. ether or distd. in vacuo gave over 65% following N,N'-bis(2-carbomethoxyethyl) derivs. (VII), (compd., b.p./mm., and m.p. dipicrate given): [MeO2C(CH2)2NRCH2]2 (VIII) (R = Me), 114-17°/0.3, 173-6°; VIII (R = Et), 125-7°/0.5, 144-8°; VIII (R = Pr), 120-5°/0.3, 153-7°; 1,4-bis(2-carbomethoxyethyl)piperazine, - (m. 53-5°), -; [MeO2C(CH2)2NEtCMe2]2 (VIIIa), 140-60°/0.3, -. The reaction with CH2:CMeCO2Me and V required more energetic conditions (20 hrs. on a water bath with a 5-molar excess of ester; addn. of some hydroquinone) and gave 20% (MeO2CCHMeCH2NMeCH2)2, b10 145-55° (dipicrate m. 167-71°), along with 30% MeO2CCHMeCH2NMeCH2CH2NHMe, b0.1 54-7° [dipicrate m. 66-70° (EtOH)]. Reaction of (H2NCH2)2 with 5 moles VI (18 hrs. on a water bath) gave 74% [(MeO2CCH2CH2)2NCH2]2 (IX), b0.6 150-70°; dipicrate m. 164-7°. VII (0.1 mole) in 60-100 ml. abs. Et2O or tetrahydrofuran (THF) added dropwise to 5.0 g. LiAlH4 suspended in 50-70 ml. abs. Et2O (or THF) with stirring and cooling, the mixt. refluxed 8-10 hrs. (1 g. LiAlH4 was added after every 2 hrs.) and decompd. with the smallest possible amt. ice H2O, the ppt. filtered off and washed with Et2O, and the combined Et2O solns. evapd. gave over 60% bis propanol compds.; the compds. were recrystd. (piperazine derivs. from MeOH) or distd. in vacuo. Redn. of IX gave poorer yields (up to .apprx.30%) [(HOCH2CH2CH2)2NCH2]2 (IXa). (4) PhCH2NH(CH2)3OH (X) (28.2 g.) dissolved in 25 g. 80% HCO2H with cooling, 17 g. 35% aq. HCHO added, the soln. heated 16 hrs. on a water bath, cooled, treated with 20 ml. concd. HCl, and evapd. in vacuo, and the viscous residue made alk. with 40% aq. NaOH under Et2O and repeatedly extd. with Et2O gave 24.0 g. PhCH2NR(CH2)3OH (XI) (R = Me), b10 143-4°. X (69.8 g.) in 60 ml.

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EtOH treated portionwise with 140 g. EtBr with cooling and the soln. kept 3 days at room temp. in a closed vessel and fractionated gave 83 g. recovered EtBr, then EtOH, and finally 64.0 g. XI(R = Et), b12 148-50°,n25D 1.5165. XI(R = Me) (24 g.)in 150 ml. EtOH hydrogenated 6 hrs. over Raney Ni at 80-90° and 50 atm. H in a stirring autoclave gave 6.6 g. RNH(CH2)3OH (XII) (R = Me), b. 175-7° n20D 1.4479. Similar hydrogenation of XI (R = Et) (100°, 100 atm. H, 12 hrs.) gave 85% XII (R = Et), b. 184-7°, n22 1.4475. CH2:CHCO2Et (22 g.) added dropwise during 60 min. to 10 g. MeNH2 in 60 ml. EtOH at 40-50° with stirring and the mixt. heated 1 hr. on a water bath gave 14.5 g. MeNH(CH2)2CO2Et, b12 60-2° (dipicrate m. 90-2°), which reduced with LiAlH4 in Et2O gave 68% XII (R = Me). XII (R = Me) (8.2 g.) heated to 120-30° with stirring, 7.0 g. (CH2Br)2 added dropwise during 25 min., and the mixt. heated and stirred 2 hrs. at 120°, cooled, dissolved in 10 ml. hot H2O, treated with 5 ml. 40% aq. NaOH, and continuously extd. with Et2O gave 3.4 g. [HO(CH2)3NRCH2]2 (XIII) (R = Me). Similarly was prepd. from XII (R = Et) [3 moles XII (R = Et)/ mole (CH2Br)2 used; heated 5 hrs. at 120°] 64% XIII (R = Et) along with 30-40% HO(CH2)3NEtCH2CH2NHEt. (CH2Cl)2 (50 g.) added dropwise during 80 min. to 150 g. H2N(CH2)3OH (XIV) heated to 110° (bath) with stirring, the mixt. stirred 4 hrs. at 110°, cooled to 40-50°, and dissolved in 500 ml. EtOH, 40 g. finely powd. NaOH added with stirring, after a time the pptd. NaCl filtered off and washed twice with 50 ml. EtOH and twice with 50 ml. MeOH, and the combined filtrates neutralized with concd. HCl, filtered, and fractionated gave 84.5 g. recovered XIV, b12 81-5°, and 53.6 g. [HO(CH2)3NHCH2]2 (XV), b0.1 155-60°, which solidified during distn. (m. .apprx.70°) and necessitated flushing the condenser with hot water [XV.2HCl m. 152-5° (EtOH); XV dipicrate m. 214-16°]; XV contained 4-5% N,N'-bis(3-hydroxypropyl)piperazine (XVI) which crystd. from MeOH at 0°. XV (39.3 g.) dissolved in 62 g. 85% HCO2H with cooling, the soln. heated on a water bath, 50 g. 35% aq. HCHO added dropwise with stirring, the soln. heated 24 hrs. and evapd. in vacuo, and the residue evapd. with H2O and EtOH, treated with 30-40 ml. 40% aq. NaOH (pH 8-9), heated 1 hr. on a water bath, and continuously extd. with Et2O (24 hrs.) gave after distn. 24.8 g. Va; Va contained 2-3% XVI. Va (25 g.) in 100 ml. Et2O treated with 40 ml. petr. ether pptd. 0.5-0.75 g. XVI, m. 135-40°. X (30.0 g.) treated dropwise at 110° with 14.0 g. (CH2Br)2 with stirring, the mixt. heated and stirred 3 hrs. at 100-10° and made alk. with concd. aq. NaOH, and the product isolated by repeated extn. with C6H6 gave 8.8 g. unchanged X, b0.4 89-101°, and then 18.5 g. [HO(CH2)3N(CH2Ph)CH2]2, b0.04 214-18°, n20D 1.5510 [dipicrate m. 200-3° (Me2CO-MeOH)]. (5) Anhyd. piperazine (8.6 g.) in 80 ml. C6H6 treated portionwise with 15.9 g. MeO2CCH2CH2COCl in 30 ml. C6H6, the mixt. kept 4 hrs. at room temp. and filtered hot, the filter cake washed repeatedly with hot C6H6, and the combined filtrates evapd. in vacuo gave 14.0 g. N,N'-bis(β-carbomethoxypropionyl)piperazine, m. 122-4° (C6H6-Et2O). Similarly were prepd. 80% N,N'-dimethyl-N,N'-bis(β-carbomethoxypropionyl)ethylenediamine (XVII), b0.05 190-200°, 82% N,N'-di-Et analog of XVII, m. 66-70°, and 76% N,N'-bis(γ-carbomethoxybutyryl)piperazine, m. 60-3° (C6H6-Et2O). Redn. of these compds. with LiAlH4 in THF (boiling 18 hrs.) gave the corresponding bis alkanols. 3,4,5-(MeO)3C6H2CO2(CH2)4Cl and V in DMF (or PhMe) heated several hrs., the soln. evapd. in vacuo, the residue partitioned between EtOAc and H2O, and the aq. phase acidified gave I; the EtOAc washed with

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dil. HCl and evapd. and the residue repeatedly recrystd. from MeOH gave .apprx.30% 3,4,5-(MeO)3C6H2CO2(CH2)nO2CC6H2(OMe)3-3,4,5 (XVIII) (n = 4), m. 118-22°, also obtained by reaction of HO(CH2)4OH and Ia in abs. dioxane in the presence of C5H5N; the acid soln., according to its chromatogram, contained a mixt. of 5 amines. From HO(CH2)3OH and Ia was similarly obtained XVIII (n = 3), m. 90-2° (MeOH-Et2O). [Me2C(NH2)]2 heated 45 min. on a water bath with 2.2 moles Ac2O in Et2O gave 86% N,N'-di-Ac deriv., m. 168-9°, which boiled 16 hrs. in THF with excess (4 moles) LiAlH4 gave 60% [Me2C(NHEt)]2, b10 71-4° [dipicrate m. 192-6° (EtOH-H2O)], which treated with VI (large excess, 15 hrs. on a water bath) gave VIIIa, reduced to the bis propanol (XVIIIa) with LiAlH4 in Et2O or THF. Redn. of (MeO2CCH2CH2)2NEt with LiAlH4 in Et2O gave 60% [HO(CH2)3]2NEt (XIX), b0.2 107-9°, n20D 1.4702. The phys. consts. of the following addnl. bis alkanols were recorded (compd., method of synthesis, b.p./mm., m.p., n20D, m.p. dipicrate given): XIII (R = Et), B-2 and B-3, 125-30°/0.5, -, -, 151-2°; XIII (R = Pr), B-3, 140-50°/0.3, -, 1.4752, 153-6°; HO(CH2)3NMe(CH2)3NMe(CH2)3OH, B-3, 120-30°/0.2, -, -, -,; [HO(CH2)4NRCH2]2, (XX) (R = Me), B-4, 160-70°/0.5, -, 148-52°; XX (R = Et), B-4, 130-40°/0.2, -, 1.4772, 114-17°; IXa, B-3, 170-95°/0.1, -, -, 190-5°; XVI, B-3, -, 142-4°, -, -; N,N'-bis(4-hydroxybutyl)piperazine, B-4, -, 114-16°, -, -; N,N'-bis(5-hydroxypentyl)piperazine, B-4, -, 110-12°, -, -; XVIIIa, B-3, 160-80°/0.2, 88-93°, -, -; (HOCH2CHMeCH2NMeCH2)2, B-3, 100-10°/0.1, -, -, 149-52°. To 8.0 g. XIX and 18 g. C5H5N in 80 ml. abs. dioxane was added dropwise during 30 min. 24.2 g. Ia in 60 ml. abs. dioxane with stirring at room temp., the mixt. heated and stirred 3 hrs. at 100° and evapd. in vacuo, the residue dissolved in EtOAc, the soln. dried and evapd., the residue dissolved in Et2O, some petr. ether added, the soln. cooled in ice overnight, the ppt. [1.4 g. [3,4,5-(MeO)3C6H2CO]2O] filtered, and the filtrate evapd. gave 23.6 g. [3,4,5-(MeO)3C6H2CO2(CH2)3]2NEt; HCl salt m. 170-5° (90% EtOH). Reaction of appropriate acid chloride with bisalkanols to bisesters was effected either (as described above) in abs. dioxane (or C6H6) with addn. of C5H5N (or another tertiary amine) or without addn. of base. The yields were between 60-90%. When base was not added, di-HCl salts of bisesters sepd. after some time. The mixt. was heated 3 hrs., dild. with Et2O, and satd. with HCl to complete the pptn. of the HCl salts. When the HCl salts did not sep. as solids, the mixt. was evapd., the residue treated with concd. aq. Na2CO3, and the product extd. with Et2O or EtOAc and converted to the HBr salt. The synthesis of the better crystg. piperazine derivs. was advantageously carried out by treating the bisalkanol with 2.1 moles acid chloride in boiling PhMe (or abs. dioxane). Satn. with HCl gave the di-HCl salt in over 60% yield. The bis(4-aminobenzoic acid esters) and bis(4-acetamidobenzoic acid esters) were obtained by hydrogenation of the corresponding NO2 compd. in EtOH over Raney Ni followed by acetylation with Ac2O. The following bisesters, 3,4,5-RR1R2C6H2CO2(CH2)n NZ(CH2)m NZ(CH2)nO2CC6H2RR1R2-3,4,5, were prepd. (method, Z, m, n, R, R1, R2, salt, m.p. salt given): by debenzylation of the corresponding PhCH2 deriv., H, 2, 3, MeO, MeO, MeO, di-HCl, 187-93°; A, Me, 2, 2, MeO, MeO, MeO, di-HCl, 173-6°; B, Me, 3, 2, H, NO2, H (XXII), -, 200-4° (decompn.) (free base); by hydrogenation of XXI, Me, 3, 2, H, NH2, H, di-HCl, above 130° (decompn.) [bis (H oxalate) m. 193-5° (decompn.)]; B, Me, 2, 3, H, H, H, di-HCl, 188-93°; B, Me, 2, 3, H, MeO, H, di-HCl, 184-9°; B, Me, 2, 3, MeO, MeO, H, di-HCl, 228-31°; B, Me, 2, 3, MeO, H, MeO, di-HCl, 134-9°; B, Me, 2, 3, Cl, MeO, H,

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di-HCl, 168-73°; A and B, Me, 2, 3, MeO, MeO, MeO (XXII), di-HCl, 170-4° (free base m. 75-7°; di-HBr salt m. 196-200°); B, Me, 2, 3, EtO, EtO, EtO, di-HBr.2H2O, 63-8°; B, Me, 2, 3, MeO, 3,4,5-(MeO)3C6H2CO2, MeO, di-HBr, 166-75°; B, Me, 2, 3, H, NO2, H, di-HCl, 204-10°; B, Me, 2, 3, H, NHAc, H, di-HCl, 149-52°; A and B, Me, 3, 3, MeO, MeO, MeO, di-HCl, 156-9°; A, Me, 4, 3, MeO, MeO, MeO, di-HCl, 193-7°; A, Me, 10, 3, MeO, MeO, MeO, di-HCl, 70-85°; B, Me, 2, 4, MeO, MeO, MeO, di-HCl, 152-7°; B, Me, 2, [(CH2)n =] CH2CHMeCH2, MeO, MeO, MeO, di-HCl, 200-3°; A and B, Et, 2, 3, MeO, MeO, MeO (XXIII), di-HBr.H2O, 76-9°; B, Et, 2, 3, EtO, EtO, EtO, di-HBr. H2O, 53-60°; B, Et, 2, 3, MeO, 3,4,5-(MeO)3C6H2CO2, MeO, di-HBr, 170-8°; B, Et, [N(CH2)n N =] NCMe2CMe2N, 3, MeO, MeO, MeO, monopicrate, 140-5°; B, Et, 2, 4, MeO, MeO, MeO, di-HBr, 182-6°; B, Pr, 2, 3, MeO, MeO, MeO, di-HBr, 200-5°; B, Pr, 2, 3, EtO, EtO, EtO, di-HBr,167-74°; B, PhCH2, 2, 3, MeO, MeO, MeO, di-HCl,97-100°; B, 3,4,5-(MeO)3C6H2CO2(CH2)3, 2, 3, MeO, MeO, MeO, di-HCl, foam. Similarly prepd. were the following 3,4,5 - RR1R2C6H2CH2CO2(CH2)nNZ(CH2)nNZ(CH2)O2CCH2C6H2RR2-R-3,4,5: A, Me, 2, 2, MeO, MeO, MeO, di-HCl, 152-60°; A and B, Me, 2, 3, H, MeO, H, di-HCl, 170-5°; B, Me, 2, 3, MeO, MeO, MeO, di-HCl, 121-7°; B, Me, 2, 4, H, MeO, H, di-HCl, 156-61°. The following piperazine derivs. (XXIV) were prepd.: (method, n, R, R1, R2, R3, m.p., salt, m.p. salt given): B, 2, H, H, MeO, H, 98-101°; di-HCl, 235-40°; B, 2, MeO, MeO, H, H, -, di-HCl, 198-204°; B, 2, H, MeO, H, MeO, -, di-HCl, 200-5°; B, 2, H, MeO, MeO, MeO, -, di-HCl, 215-18°; B, 2, H, Cl, MeO, Cl, 136-9°, di-HCl, 202-5°; A and B, 3, H, MeO, MeO, MeO (XXV), 116-19°, di-HCl, 216-20° (di-HBr salt m. 222-7°); A and B, 3, H, H, NO2, H (XXVI), -, di-HCl, 218-22°; by hydrogenation of XXVI, 3, H, H, NHAc, H, 187-90°, di-HCl, 228-33°; B, 4, H, H, MeO, H, 130-2°, di-HCl, 217-23°; B, 4, H, MeO, MeO, H, 97-100°, di-HCl, 202-6°; B, 4, H, Cl, Cl, H, 97-9°, di-HCl, 210-12°; B, 4, H, MeO, MeO, MeO, 100-4°, di-HCl, 195-200°; B, 4, H, Cl, MeO, Cl, 111-15 , di-HCl, 189-91°; B, 4, H, EtO, EtO, EtO, 88-90°, di-HCl, 197-205°; B, [(CH2)n =] CH2C.tplbond.CCH2, H, MeO, MeO, MeO, 157-9°, di-HCl, 185-93°; B, 5, H,MeO, MeO, MeO, 91-3°, di-HCl, above 200° (decompn.). Pharmacol. and clinical investigation indicate that many of the trimethoxybenzoic ester compds. (particularly XXII, XXIII, and XXV) possess, along with the expected local anesthetic and antihistaminic activity, antifibrillatory and coronary vessel dilating activity, so that they appear suitable for various cardiac diseases.

ACCESSION NUMBER: 1964:469144 CAPLUS
DOCUMENT NUMBER: **61:69144**
ORIGINAL REFERENCE NO.: 61:11994c-h,11995a-h,11996a-h,11997a-d
TITLE: Pharmacodynamically active 3,4,5-trimethoxybenzoates and related esters of symmetrical bis(hydroxyalkyl)piperazines and dialkylpolymethylenediamines
AUTHOR(S): Schloegl, K.; Schloegl, Rosemarie
CORPORATE SOURCE: Univ. Vienna
SOURCE: Monatshefte fuer Chemie (1964), 95(3), 922-41
CODEN: MOCMB7; ISSN: 0026-9247
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 61:69144

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GI For diagram(s), see printed CA Issue.
AB cf. C.A. 52, 2859f. N-(5-Bromophenyl)phthalimide (220 g.), 480 g. EtNH(CH2)2OH, and 2 l. xylene refluxed 18 hrs., cooled, treated with 1 mole K2CO3, and evaporated in vacuo, the residue extracted with CH2Cl2, the extract evaporated, the residual crude N-[5-(ethyl(2-hydroxyethyl)amino]pentyl)phthalimide treated with 200 cc. concentrated HCl and 200 cc. H2O with cooling, the mixture refluxed 4 hrs., cooled, and filtered, the residue washed with 25 cc. H2O, the combined filtrates basified strongly with saturated aqueous KOH and saturated with solid KOH, and the oily layer dried with solid KOH and distilled gave 45 g. H2N(CH2)5NEt(CH2)2OH, bl 103-4°, n25D 1.4870. AmNH(CH2)2OH (52.6 g.) treated below 30° with stirring during 7 min. with 23.4 g. CH2:CHCN (I), stirred 2 hrs. at room temperature, heated 1 hr. at 80°, kept 18 hrs. at room temperature, and stirred 2 hrs. in vacuo, the residue (72 g.) dissolved in 300 cc. EtOH (saturated with NH3), hydrogenated 1.5 hrs. at 100° and 1100 lb. over Raney Ni, and filtered, and the filtrate distilled gave 54.5 g. H2N(CH2)3NAm(CH2)2OH, b16 162-5°, n28D 1.4664. I (106 g.) added dropwise with stirring to 200 g. 3-piperidinol at 30-40° with cooling, kept 18 hrs. at room temperature, and heated 2 hrs. on the steam bath (the last hr. in vacuo) gave crude 3-hydroxy-1-piperidinepropionitrile (II). The crude II and 90 cc. Et3N hydrogenated 40 min. at 70-102° and 1775 lb. over 90 g. moist Raney Co (previously washed with absolute EtOH and cyclohexane), diluted with EtOH and cyclohexane, and evaporated in vacuo on the steam bath, and the residue distilled gave 234 g. 1-(3-aminopropyl)-3-piperidinol, b0.07 79-80°, n25D 1.5022. 7-Chlorobenz[c]acridine (III) (0.038-0.080 mole), 0.042-0.085 mole of the appropriate aminoalkylaminoalkanol, and 40-60 g. PhOH heated 3-4 hrs. on the steam bath, cooled, poured into 10-20 cc. concentrated HCl in 125-200 cc. Me3CO, cooled, and diluted with 200-400 cc. Me2CO or Et2O gave the corresponding [(benz[c]acridin-7-ylamino)alkylamino]alkanol-2HCl (IV.2HCl) (method A). III (0.030-0.038 mole) and 25-40 g. PhOH heated with stirring to 120°, cooled to 80°, treated with 0.035-0.042 mole of the appropriate diamine, heated 3 hrs. with stirring at 80-110°, poured into a solution of 5-90 g. KOH in 300-400 cc. H2O, and extracted with Et2O or CHCl3, and the extract dried, decolorized, and treated with dry HCl or an appropriate acid in Et2O gave the corresponding IV salt (method B). In this manner were prepared the following V(X, R, salt-forming acid, moles H2O of crystallization, m.p. of salt, % yield, and method given): (CH2)2, H, 2HCl, 0, 147° (decomposition) (MeOH-Et2O), 38, A; CH2CH(Me), H, 2HCl, 2, indefinite at 85° (EtOH-EtOAc), 80, A; (CH2)4, H, 2HCl, 1, 215-20°, -, -; (CH2)3, Et (VI), 2HCl, 0, 223-5° (decomposition) (MeOH-Me2CO), 52, A (57% by method B); (CH2)2, Am, 2HCl, 2.25, 150° (EtOH-EtOAc), 60, A; (CH2)5, Et, 2o-HOC6H4CO2H, 0.25, 133-5° (decomposition) (MeOH), 63, B; (CH2)2, (CH2)2OH (VII), 2HCl, 0, 220-30° (MeOH), 91, A. III (0.030 mole) and 26 g. PhOH treated slowly at 80° with 0.035 mole H2N(CH2)3N(CH2CH2OH)2, heated 3 hrs. with stirring at

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100°, cooled, and slowly poured into 90 g. KOH in 300 cc. H2O, the aq. layer decanted, the gummy residue extd. with dil. HCl, the acidic ext. basified and filtered, and the residue treated with Me2CO and C and then with dry HCl yielded 43% VII. III (0.100 mole), 0.105 mole MeCH(NH2)(CH2)3NEtCH2CH2OH, and 60 g. PhOH heated 2 hrs. with stirring at 120-30°, cooled, stirred into 20% aq. NaOH, and extd. with CHCl3, the ext. worked up, the residual oil dissolved in EtOH, acidified with alc. HCl, dild. with Me2CO-Et2O to give a dark tarry ppt., the solvents evapd., the residue basified with NH4OH and extd. with Et2O, the ext. washed and extd. with Et2O, the ext. washed, dried, and treated with alc. HCl, and the orange ppt. dissolved in MeOH, evapd., powdered, and dried 24 hrs. at room temp. yielded 45% V [X = CHMe(CH2)3, R = Et].2HCl.0.5H2O, m. 135-40°. III (55.5 g.), 40 g. MeCH(NH2)(CH2)3N(CH2CH2OH)2, and 250 g. PhOH heated 4 hrs. with stirring on the steam bath, cooled, and poured into 2 l. Me2CO contg. excess concd. HCl, the pptd. orange-red tar triturated with Me2CO and Et2O, the crude solid recrystd. twice from EtOH, the resulting dull yellow solid dissolved in H2O, the soln. poured into excess NH4OH and extd. with CHCl3, the ext. dried, concd. in vacuo to 100 cc., dild. with 1.5 l. Et2O, treated with dry HCl, and filtered, and the residue equilibrated in air gave 37% V [X = CHMe(CH2)3, R = CH2CH2OH], m. 80° (indefinite) (EtOH). V [X = (CH2)3, R = Et].2HCl.H2O (40 g.) in H2O treated with C, filtered, and basified with NH4OH, the aq. soln. decanted, and the residue treated with Et2O yielded 28 g. VI, m. 80-2° (decompn.) (MeOH). By method B was prepd. in the usual manner 3-(benz[c]acridin-7-ylamino)propylaminopropanol, m. 70° (indefinite). III (20 g.), 12.7 g. Et2NCH2CH(OH)CH2NH2, and 75 g. PhOH heated 2 hrs. with stirring at 110°, kept 16 hrs. at room temp., poured with stirring into 175 g. KOH in 1 l. H2O contg. 500 g. ice, and extd. with Et2O, the ext. washed, decolorized, dried, and treated with dry HCl, and the ppt. washed with Et2O and Me2CO and dried 24 hrs. at 40° in vacuo gave 14.2 g. (benz[c]acridin-7-ylamino)-3-diethylamino-2-propanol, hygroscopic yellow powder, m. 100° (indefinite). III (26.3 g.), 16.6 g. II, and 50 g. PhOH heated 3 hrs. with stirring on the steam bath, cooled, treated with excess alc. HCl, dild. with 1 l. dry Me2CO, chilled, and filtered, the residue washed with dry Me2CO, added to excess NH4OH, and extd. with CHCl3, the ext. washed, decolorized, and evapd. in vacuo, the oily residue dissolved in abs. EtOH, treated with excess alc. HCl, stirred and warmed with MeOH, dild. with dry Me2CO, chilled, and filtered yielded 42.5 g. 1-[3-(benz[c]acridin-7-ylamino)propyl]-3-piperidinol-2HCl.H2O, m. 262°. The appropriate IV.2HCl (0.009-0.076 mole) dried in vacuo 18-48 hrs. at 35-100°, stirred and heated 7-24 hrs. on the steam bath in 40-500 cc. of the appropriate acid chloride, kept 1-16 hrs. at room temp., dild. with Et2O, and filtered, and the residue dried in vacuo at room temp. 5-18 hrs. and recrystd. gave the corresponding esters of IV (method C). IV.2HCl (0.011-0.034 mole) dried 18 hrs. at 35-100°, heated 20-4 hrs. with 0.011-0.040 mole succinic anhydride at 100-50° under N, the mixt. dissolved in boiling abs. EtOH, the soln. dild. with dry Et2O, and the yellow ppt. filtered off, washed with Et2O, and dried gave the ester of IV (method D). In this manner were prepd. the following esters of V.2HCl (X, R, ester, moles H2O of crystn., m.p., % yield, and method given): (CH2)2, H, acetate, 0.5, 135-45°, 100, C; (CH2)2, H, palmitate, 1, 170-80° (MeOH), 67, C; (CH2)2, H, 0.5, H succinate, 80° (decompn.), 77, D; (CH2)3, Et, acetate, 1, 174-6°, 93, C; (CH2)3,

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Et, heptanoate, 2, 147-9°, 80, C; (CH2)3, Et, palmitate, 3, 168-71°, 92, C; (CH2)3, Et, H succinate, 1, 120° (HCONMe2-Et2O), D (under N); (CH2)3, (CH2)2OH, H succinate, 0.25, 80° (decompn.), 90, D; (CH2)3, (CH2)2OCOC15H33, palmitate, 2.5, 185-6° (MeOH), 85, C. III (10 g.) and 40 g. PhOH heated 15 min. with stirring on the steam bath, heated 2 hrs. with stirring with 6.1 g. H2N(CH2)3NH(CH2)2OH on the steam bath, poured into 500 cc. 10% aq. NaOH, and extd. with Et2O, the ext. washed, dried, and treated dropwise with 2.9 g. (CH2COCl)2 with shaking, and the mixt. kept 1 hr. at room temp., treated with dry HCl, and filtered gave 16 g. di-2-[[3-(benz[c]acridin-7-ylamino)propyl]ethylamino]ethyl succinate-HCl.3H2O, m. 130-5° with softening at 80°.

ACCESSION NUMBER: 1958:55920 CAPLUS
DOCUMENT NUMBER: **52:55920**
ORIGINAL REFERENCE NO.: 52:10082i,10083a-i,10084a-g
TITLE: Synthetic amebicides. IV. [(Benz[c]acridin-7-ylamino)alkylamino]alkanols and their esters
AUTHOR(S): Elslager, Edward F.; Short, Franklin W.; Sullivan, Marie Jo; Tendick, Frank H.
CORPORATE SOURCE: Parke, Davis & Co., Detroit, MI
SOURCE: Journal of the American Chemical Society (1958), 80, 451-5
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 52:55920

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AB cf. preceding abstract For pharmacologic study methylated and acylated derivs. of Ph2C(CH2NH2)CH2NR2 (I) are prepared The attempted reduction of the CN group in Ph2C(CN)CH2NR2 (II) by catalytic hydrogenation gives 75% of the cleavage product Ph2CHCH2NH2 from II (R = Me), whereas Na in EtOH on II (R = Me or Et) causes cleavage at a different point to give Ph2CHCH2NR2, 65 and 57% yields, resp. Ph2CHCH2NEt2, b0.4 121-2°, n25D 1.5499; hydrochloride, m. 155-6° (from Me2CO). Successful reduction without cleavage requires 2 moles LiAlH4 to 1 mole II [cf. C.A. 43, 1280f; Amundsen and Nelson, C.A. 45, 6569g; Nace and Smith, J. Am. Chemical Society 74, 1861(1952)]. By otherwise standard LiAlH4 reduction of II, derivs. of I are prepared (NR2, % yield, n24D, b.p., and m.p.): NMe2(Ia), 86.5, 1.5737, b0.2 126-8°, 37-9° (from C5H12) [di-HCl salt, m. 242-4° (decomposition) (from MeOH-Et2O)]; 1-pyrrolidinyl (Ib), 82.5, -, -, 54-5° (from Skellysolve B); piperidino (Ic), 71, 1.577, -, - [monoformate, m. 148-9° (from MeOH-Et2O)]. Ia (10.16 g.) is methylated with 3.0 g. paraformaldehyde in 80 cc. 90% HCO2H to yield 9.1 g. Ph2C(CH2NMe2)2, b0.1 110-14°, n25D 1.5579; monomethiodide, m. 148-9° (decomposition) (from MeOH-Et2O); dimethiodide, m. 203-4° (decomposition) (from MeOH). Well known methods are used to prepare other derivs., Ph2C(CH2NR2)CH2NHCOR' (III), from Ia, b, and c, (cf. Human and Mills, C.A. 43,1336a). From Ia, R' and m.p. are: H, 113-14°; Me, 97-8°; Et, 80-1°; Pr, 69-71°; PhCH2, 86-8°; Ph, 95-6° [hydrochloride, m. 228-9° (decomposition)]; o-ClC6H4, - [hydrochloride, m. 249-50° (decomposition)]; m-ClC6H4, - [hydrochloride, m. 211-12° (decomposition)]; p-ClC6H4, - (hydrochloride, m. 160-3°); OEt,- hydrochloride, m. 216-17° (decomposition)]; NH2, 175-7°; from Ib: H, 112-13°; Me, 86.5-7.5°; Et, 94-6°; Pr, 79-80°; PhCH2, 88-90°; Ph, 152-3°; OMe, - [hydrochloride, m. 215-16° (decomposition)]; OEt, - (hydrochloride, m. 209-10° (decomposition)]; NH2, 151-2°; from Ic; H, 96-7°; Me, 119-20°; Et, 89-90.5°; Pr, 72-4°; PhCH2, 80-1°; Ph, 133-4°; OMe, - [hydrochloride, m. 227-8° (decomposition)]; OEt, - [hydrochloride, m. 197-8° (decomposition)]; NH2, 161-3°. A few compds. III show strong but brief analgesic action, and nearly all have local anesthetic activity.

ACCESSION NUMBER: 1954:3381 CAPLUS
DOCUMENT NUMBER: 48:3381
ORIGINAL REFERENCE NO.: 48:581g-i,582a-c
TITLE: 2,2-Diphenyl-1,3-propanediamines and N-substituted derivatives
AUTHOR(S): Zaugg, Harold E.; Horrom, Bruce W.
CORPORATE SOURCE: Abbott Labs., Chicago
SOURCE: Journal of the American Chemical Society (1953), 75, 292-4
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 48:3381

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AB cf. C.A. 41, 1609h In unpublished work of Longinov and Filippov (1935) it was found that when the Na used for the reduction of esters contains even as little as 0.01% of K the yields of the alcs. drop rapidly. In the reduction of BuCH(CO2Et)2 the yield is zero when the Na contains 0.1% K. However, if the amount of K is increased, the yields rise again and become normal with 2% K. This is important since Na is usually contaminated with traces of K. This study was extended to the reduction of Et2NCH2CH2CN and 1-piperidinepropionitrile in BuOH by the method of Suter and Moffett (C.A. 28, 1659.1). Tech. Na gave 38-50% of the diamine, pure Na 51-63%, while tech. Na with the addition of 2% K gave 65.9-70% diamine, in the case of Et2NCH2CH2CN. With the piperidyl compound the yields were 42-46%, 48.9-57.2%, and 48-57%, resp., thus indicating a similarity to the results obtained in ester reduction. The high-K Na gave reproducible yields and tended to give smaller amts. of higher-boiling products (presumably secondary amines). The Rupe technique (immediate steam distillation) gave similar results, but was inconvenient because of the low volatility of the products.

ACCESSION NUMBER: 1948:618 CAPLUS
DOCUMENT NUMBER: 42:618
ORIGINAL REFERENCE NO.: 42:112a-d
TITLE: Syntheses with acrylonitrile. IV. Reduction of nitriles with sodium in alcohol medium
AUTHOR(S): Kost, A. N.; Terent'ev, A. P.
CORPORATE SOURCE: Moscow State Univ.
SOURCE: Zhurnal Obshchei Khimii (1947), 17, 105-8
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Russian
OTHER SOURCE(S): CASREACT 42:618

L4 ANSWER 69 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C.A. 30, 7551.1. The preparation of various N-substituted 2-methoxy-5-chloro-9-aminoacridines by 2 methods is described. 2-Carboxy-4-methoxy-6'-chlorodi-phenylamine (I), prepd in 75% yield from 2,5-Br(MeO)-C6H3CO2H and o-chloroaniline, m. 186-8° when recrystd. from glacial AcOH. I and POCl3 gave 85% 2-methoxy-5,9-dichloroacridine (II), m. 157-8° (from EtOAc). I and PCl5 gave 70% 2-(6-chloroanilino)-5-methoxybenzoyl chloride (III), m. 112° (from low-boiling absolute petr. ether). The following requisite amines were prepared in the yields shown by reduction, with Na and absolute alc., of the nitrile obtained from acrylonitrile and the appropriate secondary amine: 3-diethylaminopropyl (IV), 78%; 3-dibutyl-aminopropyl, 75%; 3-diamylaminopropyl, 68%; and 3-piperidinopropyl, 88% (cf. C.A. 38, 3617.3). II (3 g.) and 1.5 g. IV were heated in 12 g. phenol 3 h. at 100-10° poured into excess 2 N NaOH, extracted with ether, the ether solution extracted with 25 cc. 5% AcOH, and the aqueous layer filtered, decomposed with alkali, and extracted with ether, from which the 2HCl salt of 2-methoxy-5-chloro-9-(3-diethylamino-propylamino)acridine (V) was obtained in 50% yield by the addition of alc. HCl. V was also obtained in 75% yield by refluxing III and IV in C6H610 min., removing the C6H6 in vacuo, refluxing the residue with POCl3 2 h., removing the excess POCl3 under vacuum, decomposing the aqueous solution of the residue with alkali, extracting with ether, and precipitating V as above. 2-Methoxy-5-chloro-9-(4-diethylaminobutyl-amino)acridine-2HCl(3-diethylaminobutylamino in original, apparently an error), m. 245-7°, was obtained in a similar fashion from II or III and 4-diethylaminobutylamine. 2-Methoxy-5-chloro-9-(5-diethylaminoamylamino) acridine dipicrate, m. 180-2° (from absolute alc.), was prepared from II and 5-diethylaminomylamine. The following 9-substituted 2-methoxy-5-chloroacridine-2HCl were prepared in a similar manner and recrystd. from a mixture of absolute alc. and ether: (3-dipropylaminopropylamino), m. 308-10°; (3-di-butylaminopropylamino), m. above 310° (decomposition); (3-diamylaminopropylamino), m. 325-7° (decomposition); (3-piperidinopropylamino), m.253-5° (decomposition). Analogous prepns. of 2-methoxy-5-chloro-9-(p-sulfanilamidophenylamino)acridine, m. 250-1° and 2-methoxy-5-chloro-9-(p-arsonophenylamino)acridine, m. 257-9° are described. II and HCl gave 2-methoxy-5-chloroacridine, m. 269-70°. II and (NH4)2CO3 heated in phenol at 130° 15 min., cooled, diluted with ether, and dry HCl gas passed into the solution caused 2-methoxy-5-chloro-9-amino-acridine-HCl (VI) to precipitate After separation and purification, VI was decomposed with alkali to the free base, m. 230-2° (from alc. and acetone).

ACCESSION NUMBER: 1947:11861 CAPLUS
DOCUMENT NUMBER: 41:11861
ORIGINAL REFERENCE NO.: 41:2419h-i,2420a-e
TITLE: Acridines. I. N-Substituted 2-methoxy-5-chloro-9-aminoacridines
AUTHOR(S): Singh, Gurbakhsh; Singh, Mahan
CORPORATE SOURCE: Univ. Punjab, India
SOURCE: J. Indian Chem. Soc. (1946), 23, 224-8
DOCUMENT TYPE: Journal

L4 ANSWER 69 OF 70 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 41:11861

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AB The basically substituted propionitriles are best obtained by the addition of NH3 or primary or secondary amines to CH2:CHCN (I); γ-aminobutyronitriles were prepared from Cl(CH2)4NH2 and secondary amines; the yields are greatly improved by the use of a solvent. The mechanism by which the addition takes place probably involves a typical 1,4-addition Thus β-(3-diethylaminopropylamino)propionitrile (b25 163-5°, n20D 1.4573; picrate, m. 123°) results in 79.4% yield from I and H2N(CH2)3NEt2 and in 76.4% yield from Br(CH2)2CN and H2N(CH2)3NEt2. The reaction is reversible. When the higher β-dialkylaminopropionitriles are heated near their b. p. for any length of time, they slowly decompose to give some of the R2NH. (HOCH2CH2)2NH (II) and I, allowed to stand 8 h. at room temperature, give (HOCH2CH2)2NCH2CH2CN (III), which could be isolated as the picrate, m. 108-9°; however, attempted distillation gave 67% of III and a nondistillable polymer. Reduction of III in EtOH at 105-10° and 133 atmospheric gives 40% of 3-diethanolaminopropylamine, b2 158°, n 1.4975 (all n are n20D) (picrate, m. 156.5-7.5°). H2NCH2CH2CN (IV) (b5 66-9°, n 1.4396; picrate, m. 178°), on standing, gives NH3 and a polymer, best explained by its disproportionation into NH3 and I. The rate of addition of amines to I is discussed; this is not a function of the basic strength of the amines but is more dependent upon the size and shape of the entering mol. In certain cases a catalyst is necessary to induce reaction. In the reaction of I with NH3, NH(CH2CH2CN)2 (V) (b4 165°, n 1.4640) and N(CH2CH2CN)3 (VI) predominate when NH4OH is used but with a 7-mol. excess of liquid NH3 at 40°, the products are 22% of IV and 64% of V, with very little VI. Thus V adds to I approx. 20 times faster than NH3. I (424 g.) and 1000 g. EtNH2, warmed for 24 h. at 50° and allowed to stand 2 days at room temperature give 97% of Et2NCH2CH2CN (VII), b735 196°, n 1.4356; picrate, m. 85°; by using mol. ratios and heating on the steam bath for 8 h., the yield is 74%; 5 mols. of EtNH2 and 9.5 mols. of com. I, refluxed 0.5 h. and kept at 0° overnight, give 93% of VII. In general, better yields result if the reaction product is allowed to reach equilibrium at room temperature before distillation; the product should be distilled more or less rapidly at a low temperature if possible. β-Ethylaminopropionitrile, b30 92°, n 1.4318, 90.4%; picrate, m. 163°. Bis(2-cyanoethyl)ethylamine, b30 200-2°, n 1.4591, 60%; picrate, m. 170°. Pr2NCH2CH2CN, b20 116°, n 1.4381, 88%; picrate, m. 111°. Bu2NCH2CH2CN, b20 141°, n 1.4423, 91%; picrate, m. 75°. β-Diamylaminopropionitrile, b19 159-61°, n 1.4457, 89%; the picrate is an oil. β-Dihexylaminopropionitrile, b2 145-6°, n 1.4483, 85%. β-(1-Piperidyl)-2-propionitrile, b30 129-30°, n 1.4697, 93°; picrate, m. 160°. β-(4-Morpholinyl)propionitrile, b20 149°, n 1.4710, 95%; picrate, m. 139.5°. Bis(2-cyanoethyl)ethanolamine picrate, m. 137-8°. 1-Diethylamino-3-[bis(2-cyanoethyl)amino]propane, b25 233-5°, n 1.4709, 8.8%; picrate, m. 166-7°. I and (Et2NCH2CH2CH2)2NH with 0.1 g. Cu bronze, heated at 100° for 24 h. and allowed to stand 24 h. at room temperature, give 78% of β-[bis(3-diethylaminopropyl)amino]propi

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morpholinyl)butyl]amine, b3 200-2°, n 1.4900, 23.8%; picrate, m. 136°. Bis(3-aminopropyl) ether, b32 113°, n 1.4618, 29%. 3-(3-Diethylaminopropylamino)propylamine, b25 142-4°, n 1.4630, 51%; picrate, m. 197-8°; bis[3-(3-diethylaminopropylamino)propyl]amine, b25 253-60°, n 1.4710, 31%; picrate, m. 197°. 3-[Bis(3-diethylaminopropyl)amino]propylamine b3 155-65°, n 1.4662, 52%; picrate, m. 162.5°. 3-[2-(4-Morpholinyl)ethylamino]propylamine, b2 120-3°, n 1.4870, 57.5%; picrate, m. 208°. 3-[3-(4-Morpholinyl)propylamino]propylamine, b1.5 137-40°, n 1.4878, 45.2%; picrate, m. 205°. 3-(2-Diethylaminoethoxy)propylamine, b25 118-22°, n 1.4498, 56.7°. Bis[3-(2-diethylaminoethoxy)propyl]amine, b3 175°, n 1.4582, 23.8%. 3-(3-Diethylaminopropoxy)propylamine, b25 130-2°, n 1.4500, 57.4%. Bis[3-(3-diethylaminopropoxy)propyl]amine, b3 182°, n 1.4581, 28.2%. 3-(4-Diethylamino-1-methylbutoxy)propylamine, b2 80-3°, n 1.4492, 50.5%; picrate, m. 88-9°. Bis[3-(4-diethylamino-1-methylbutyoxy)propyl]amine, b3 210-15°, n 1.4580, 23%. 3-(Methylphenylamino)propylamine, b40 171-2°, 63%; HBr salt, m. 120°; picrate, m. 189°. 9-(3-Aminopropyl)carbazole, b3 228°, 70.5%; HCl salt, m. 273°; picrate, m. 206-7°. 1-(3-Aminopropylamino)tetrahydroquinoline, b3 132-5°, n 1.5828, 82%. The following N1-sulfanilamide derivs. were prepd. through the N4-Ac derivs.; in some cases the Ac derivs. were viscous sirups which did not crystallize. 3-Diethylaminopropyl, m. 109-10°, 20%; 3-dipropylaminopropyl, m. 98-8.5°, 57°; 3-dibutylaminopropyl, HCl salt, m. 110-15°, 53.5%; 3-(1-piperidyl)propyl, m. 105.5-6°, 63.5% (Ac deriv., m. 109-11°); 3-(4-morpholinyl)propyl, m. 94.5-5°, 79% (Ac deriv., m. 97-8°); bis(3-diethylaminopropyl), HCl salt, m. 195-7°, 66.5% (Ac deriv., m. 83-5°); bis[3-(1-piperidyl)propyl], m. 74-6°, 71%. It was not possible to use C5H5N or C5H5N-Me2CO as solvents in the prepn. of these derivs.

ACCESSION NUMBER:	1944:24921 CAPLUS
DOCUMENT NUMBER:	38:24921
ORIGINAL REFERENCE NO.:	38:3617b-i,3618a-i,3619a-c
TITLE:	Basically substituted aliphatic nitriles and their catalytic reduction to amines
AUTHOR(S):	Whitmore, Frank C.; Mosher, Harry S.; Adams, Robert R.; Taylor, Robert B.; Chapin, Earl C.; Weisel, Charles; Yanko, Wm.
SOURCE:	Journal of the American Chemical Society (1944), 66, 725-31 CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE:	Journal
LANGUAGE:	Unavailable
OTHER SOURCE(S):	CASREACT 38:24921

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onitrile, b3 153°, n 1.4640; picrate, m. 157-8°. β-[2-(4-Morpholinyl)ethylamino]propionitrile, b20 183°, n 1.4817, 81.5%; picrate, m. 176.5°; β-[3-(4-morpholinyl)propylamino]propionitrile, b9 178-80°, n 1.4819, 76%; picrate, m. 148-9°. β-(2-Diethylaminoethoxy)propionitrile, prepd. in 80% yield by adding 100 g. I to 220 g. Et2NCH2CH2OH and 2.3 g. MeONa during 0.5 h. at 25°, allowing to stand overnight, treating with 4.2 mL. concd. H2SO4 and distg. the filtrate, b25 145°, n 1.4430; picrate, m. 75°; β-(3-diethylaminopropoxy)propionitrile, b25 148-50°, n 1.4440, 75.4%. β-(4-Diethylamino-1-methylbutoxy)propionitrile, b3 125-30°, n 1.4456, 66%. I (27 g.) and 53 g. PhNHMe do not react when heated at 180° for 4 h.; in the presence of 1 g. CuSO4.5H2O there results 20 g. of β-(methylphenylamino)propionitrile, b20 175-7°; no reaction occurs with PhCH2NMe3OH in dioxane at 100°; picrate, m. 118°. I (250 mL.) and 167 g. carbazole, cooled in an ice-bath and treated with 2 cc. 40% PhCH2NMe3OH, with heating on the steam bath for 1 h., give 85.4% of 9-(2-cyanoethyl)carbazole, m. 155.5°. I and tetrahydroquinoline did not react with the usual catalysts at 160° after 4 h.; addn. of 100 g. I to 133 g. of tetrahydroquinoline at 125°, with refluxing for 6 h., gives 75.5% of 1-(2-cyanoethyl)tetrahydroquinoline, b10 192°, n 1.5780; picrate, m. 172°. γ-Diethylaminobutyronitrile, b21 101-3°, n 1.4351, 86%; picrate, m. 69-70°; γ-(1-piperidyl)butyronitrile, b25 127-9°, n 1.4653, 87%; picrate, m. 117°; γ-(4-morpholinyl)butyronitrile, b25 148-50°, n 1.4665, 70%; picrate, m. 152-3°. The redn. of the nitriles to amines was carried out with approx. 10 g. Raney Ni per mol. of nitrile at temps. of 90-130° and H pressures of 67 to 270 atm. The formation of secondary amines (5-32%) was obsd., being greater in the case of the γ-aminobutyronitriles; the redn. can be controlled and the yield of primary amine increased by carrying out the hydrogenation in the presence of NH3; the yield of secondary amines can be raised by reducing the nitrile in the presence of an excess of the primary amine. It is believed that the yields of primary products can be increased in almost every case if H pressures of 250-300 atm. are employed and larger amts. of NH3 are introduced. The original gives the pressure, temp., time and solvent used for the various redns. The following compds. are reported. (CH2)3(NH2)2, b735 138°, n 1.4600, 23%; picrate, m. 178°. Et2N(CH2)3NH2, b735 168°, n 1.4355 (picrate, m. 194°), yields up to 72%; bis(3-diethylaminopropyl)amine, b3 107°, n 1.4541 (picrate, m. 153-4°), yields up to 29%. 3-Ethylaminopropylamine, b735 156°, n 1.4441, 74%; picrate, m. 193°. Bis(3-aminopropyl)ethylamine, b20 135°, n 1.4709, 16%; picrate, m. 197-9°. Pr2N(CH2)3NH2, b20 94°, n 1.4435, 49%; picrate, m. 181°; Bu2N(CH2)3NH2, b20 121°, n 1.4462, 32%; picrate, m. 188°. 3-(1-Piperidyl)propylamine, b730 205°, n 1.4750, 68.5%; picrate, m. 209-10°. Bis[3-(1-piperidyl)propyl]amine, b2 153°, n 1.4916, 10%; picrate, m. 193°. 3-(4-Morpholinyl)propylamine, b733 219°, n 1.4762, 70.6%; picrate, m. 166°. Bis[3-(4-morpholinyl)propyl]amine, b5 185°, n 1.4918, 10%; picrate, m. 213-15°; in the prepn. of the primary amine, up to 35% morpholine are formed under certain conditions. 4-Et2N(CH2)4NH2, b18 85-8°, n 1.4462, 51%; picrate, m. 155-6°. 4-(1-Piperidyl)butylamine, b25 118-20°, n 1.4756, 53.8%; picrate, m. 160.5°. Bis[4-(1-piperidyl)butyl]amine, b25 220-5°, n 1.4898, 32%; picrate, m. 202-3°. 4-(4-Morpholinyl)butylamine, b20 122°, n 1.4760, 62%; picrate, m. 148°. Bis[4-(4-

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=>
=>    ` logoff y
` IS NOT A RECOGNIZED COMMAND
The previous command name entered was not recognized by the system.
For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).

=> logoff y
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FULL ESTIMATED COST                                   198.55     568.64

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)          SINCE FILE      TOTAL
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